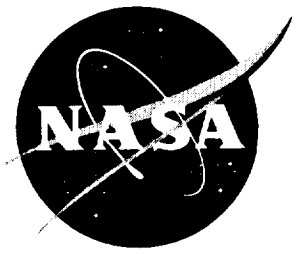

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Volume II

The Microgravity Research Experiments (MICREX) Data Base

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*Note: The seven indexes contained in Volume 1 (Indexes A to G) can be used to quickly identify critical information detailed throughout Volumes 2, 3, and 4. Information on how to use the indexes can be found in Volume 1 on page 71.

Experiment Listing: Chapters 1-11

Chapter 1
Biotechnology

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 1, Cont.
Biotechnology

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 1, Cont.
Biotechnology
Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

<Note: Experiments related to Protein Crystal Growth can be found in Chapter 16.>

<Note: An experiment entitled Aggregation of Red Blood Cells (STS 51-C, 1985) by **Principal Investigator L. Dintenfass** (Kanematsu Institute, Department of Medical Research, Sydney, Australia) should have been included in this Chapter. Information on the experiment was discovered after this Technical Memorandum had been prepared. Details of this experiment should appear in later versions of the MICREX.>

Chapter 2
Capillarity

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 2, Cont.
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Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 3
Combustion Studies

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

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Listed Primarily Chronologically,
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Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

<Note: Three additional foaming experiments can be found in Chapter 2: Capillarity.>

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Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

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Listed Primarily Chronologically,
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Containerless Processing
Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

<Note: Semicontainerless float zone experiments can be found in Chapter 9: Crystal Growth From the Melt. Other experiments related to Containerless Processing Applications (such as the creation and acoustic positioning of liquid bubbles and the creation and stability of liquid zones) can be found in Chapter 12: Fluid Physics and Chapter 2: Capillarity.>

Chapter 6, Cont.
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Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 7
Critical Point Phenomena
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Crystal Growth From Solution
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Chapter 8, Cont.
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Chapter 9
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but with Investigative Series of Principal Investigators Intact

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Crystal Growth From the Melt
Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 9, Cont.
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Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 10
Crystal Growth From Vapor

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 10, Cont.
Crystal Growth From Vapor

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 11
Diffusion

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

Chapter 11, Cont.
Diffusion

Listed Primarily Chronologically,
but with Investigative Series of Principal Investigators Intact

<Note: A related diffusion experiment entitled "Thermal Conductivity of Electrically Non-Conducting Liquids" can be found in Chapter 15: Physical Chemistry (See Aalto, MASER 1).>

CHAPTER 1

BIOTECHNOLOGY

Principal Investigator(s): McKannan, E. C. (1)
Co-Investigator(s): Griffin, R. N. (2)
Affiliation(s): (1) During Apollo: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama, Currently: Boeing Aerospace and Electronics, Huntsville, Alabama; (2) During Apollo 14: General Electric Company, King of Prussia, Pennsylvania, Currently: Unknown

Experiment Origin: USA
Mission: Apollo 14
Launch Date/Expt. Date: February 1971 (during lunar flyback)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Apollo Command Module Payload
Processing Facility: Apollo Electrophoresis Demonstration Unit (static column electrophoresis system equipped with a photographic observation system)
Builder of Processing Facility: General Electric, King of Prussia, Pennsylvania

Experiment:
Electrophoretic Separation

Most particles dispersed within an aqueous solution (or buffer) acquire either a positive or negative net surface charge. In the presence of an electric field, these charged particles migrate at different velocities toward the oppositely charged electrode. Because the particles migrate at different rates, they can be separated from one another; the process is known as electrophoresis. In the low-gravity environment, thermal convective effects (attributed to the heat generated by the electric field) and sedimentation effects (attributed to the settling of high density particles out of the sample medium) are greatly reduced. Thus, more efficient separation is expected.

In an effort to explore such separation, electrophoresis experiments were performed on the Apollo 14 mission. The electrophoresis chamber contained two electrodes (one configured at each end) and three glass separation tubes.

A mixture of two dyes ("Brilliant Blue and Amaranth Red") was selected for the first separation tube. It was suspected that while the intense color of the dyes would facilitate separation detection, the mobility of each would permit separation within the limited confines of the tube.

Hemoglobin was selected for the second separation tube. Reportedly, "It is a naturally occurring material of biological origin and closely akin to materials of practical interest for electrophoresis." (1, p. 4)

Salmon sperm DNA was selected for the third separation tube. This very high molecular weight material had been electrophoresed by Earth-based molecular methods but only with limited success. (A reaction between DNA and DANSYL chloride results in the fluorescent tagging of the DNA allowing it to be observed using a "black light.")

During the experiment, injection of the samples into the tubes occurred simultaneously. Photographs documented the subsequent sample separation.

Reportedly, "The red and blue dyes separated as expected, but no action was seen in the hemoglobin or DNA." (1, p. 5) When space separation of the dye was compared to similar ground-based separation, there were great differences in the shapes of the "boundaries." On Earth, the boundaries tend to stratify under the influence of electroosmosis, convection, etc. In space, the boundaries were sharper; "..no lateral motion of the fluid is evident which can be attributed to thermal convection or sedimentation." (1, p. 5) Interestingly, "...the velocity of the dyes in the space experiment was less than half as great as the velocity measured on earth. It is extremely unlikely that this difference represents a fundamental change in the process in the absence of gravity." (1, p. 6) Instead, it was surmised that the velocity difference resulted from a major change in the pH electrolyte which occurred between the loading of the samples and the retrieval of the payload 11 weeks later.

It was also surmised that "The hemoglobin and DNA may have been consumed during storage by bacterial action which changed the pH of the electrolyte." (1, p. 7)

Key Words: Biotechnology, Electrophoresis, Aqueous Solutions, Separation of Components, Suspension of Particles, Particle Dispersion, Particle Mobility, Particle Migration, Particle Motion, Biological Cells, Electric Field, Electrodes, Surface Charge, Thermal Convection, Sedimentation, Electroosmosis, Wall Effect, Electrolytes, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Contamination Source, Medical Applications, Biological Materials Processing

Number of Samples: three
Sample Materials: Sample 1: mixture of red and blue dye; Sample 2: hemoglobin; Sample 3: salmon sperm deoxyribonucleic acid (DNA). Buffer Materials: unknown

Container Materials: glass tubes

Experiment/Material Applications:
A study contract by the General Electric Company identified electrophoretic processing as "...one of the simplest processes in space to perform with the greatest potential benefit to mankind." (1, p. 1) It was theorized that after the advantage of electrophoretic separation was demonstrated in space (reduced convective and sedimentary effects) "...small but significant quantities of biological materials such as vaccines, viral insecticides, and other valuable materials could be purified and separated in space, economically." (1, p. 1)

References/Applicable Publications:
(1) McKannan, E. C., Krupnick, A. C., Griffin, R. N., and McCreight, L. R.: Electrophoresis Separation in Space-Apollo 14. NASA TM X-64611, 1971. (post-flight)

(2) Hannig, K. and Wirth, H.: Free Flow Electrophoresis in Space. COSPAR, Symposium on Material Sciences in Space, Philadelphia, Pennsylvania, June 9-10, 1976. In Materials Sciences in Space with Application to Space Processing, New York, American Institute of Aeronautics and Astronautics, Inc., 1977, pp. 411-422. (includes information on electrophoresis experiments during Apollo 14, 16, and ASTP; post-flight)

(3) Snyder, R. S., Bier, M., Griffin, R. N., Johnson, A. J., Leidheiser, H., Micale, F. J., Ross, S., Vanderhoff, J. W., and Van Oss, C. J.: Free Fluid Particle Electrophoresis on Apollo 16. Separation and Purification Methods, Vol. 2, 1973, pp. 259-282. (post-flight; briefly discusses Apollo 14 results)

(4) Input received from Principal Investigator E. C. McKannan, June 1993.

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Principal Investigator(s): Snyder, R. S. (1)
Co-Investigator(s): Griffin, R. N. (2)
Affiliation(s): (1) National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (2) During Apollo 16: General Electric Company, Valley Forge, Pennsylvania, Currently: Unknown

Experiment Origin: USA
Mission: Apollo 16
Launch Date/Expt. Date: April 1972
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Apollo Command Module Payload (the experiment was secured to a storage locker)
Processing Facility: Apollo Electrophoresis Demonstration Unit (Basic elements of the Apollo 14 Demonstration Unit (see McKannan, Apollo 14) were reused for this flight)
Builder of Processing Facility: Design, manufacture and testing of the flight and backup flight hardware were completed by the Space Sciences Laboratory of the General Electric Company, Valley Forge, Pennsylvania

Experiment:
Electrophoresis Demonstration

In the presence of an electric field, charged particles in solution can be separated according to their net surface charge. This method of separation, known as electrophoresis, should be enhanced in the low-gravity environment where thermal convection and cell sedimentation effects are reduced.

This Apollo 16 experiment, which was an extension of an electrophoresis investigation performed during Apollo 14 (see McKannan, Apollo 14 (this chapter)), was the first in a series of experiments designed by Snyder et al. to study reduced gravity electrophoresis. The specific objective of the investigation was to electrophoretically separate monodisperse polystyrene latex particles (0.8 microns and 0.2 microns).

The electrophoresis chamber contained platinum electrodes and three 0.64 cm diameter, 10 cm long polycarbonate (Lexan™) separation tubes. Prior to the launch of the Apollo 16 mission, polystyrene latex particles were retained in containers at one end of each of the tubes. Diameters of these containers were smaller than the tubes "..so that electrophoresis of the particles would take place down the center of the...[tubes] and away from the walls, where the electroosmotic flow tends to reverse the direction of the particles' migration." (1, p. 268)

During the experiment, a film enclosing the retaining containers was removed, and the samples were exposed to the dilute borate buffer and the imposed electric field. Initiation of the samples in all three tubes was done simultaneously, and the particles were allowed to electrophoretically migrate to the anode.

The first photograph taken of the cells (less than 0.5 cm from the sample input) demonstrated that "...the front of each group of particles was already parabolic. Presumably, the electroosmotic flow pattern of the buffer in the electrophoresis tubes quickly modified the shape of the particle bands into parabolas." (1, p. 273) Reportedly, such parabolic behavior was not observed in similar ground-based testing where sedimentation and convection effects were prominent. Additional photographs documenting the motion were taken every 20 seconds.

The first (Apollo 16) tube, which contained a mixture of 0.2 and 0.8 micron particles, experienced particle separation. "Careful exposure of the flight original negatives revealed that the nose of the combined band was much less dense..." (1, p. 274) This "less dense" region corresponded to the larger particles whose higher electrophoretic mobility had drawn them more quickly toward the anode.

The second tube was configured to separate only 0.8 micron particles, and the third tube was configured to separate only 0.2 micron particles. After examining the results concerning all three tubes, the following was reported:

"1. The actual electrophoretic mobility of the 0.2μ polystyrene latex in the borate buffer was 6.5μ cm/volt sec, in the Apollo 16 experiment.

"2. The actual electrophoretic mobility of the 0.8μ polystyrene latex in the borate buffer was 9.2μ cm/volt sec, in the Apollo 16 experiment.

"3. The potential gradient in each channel must have been reduced by approximately 40%, probably because of the presence of bubbles [in the tubes].

"4. Electroosmosis at the polycarbonate/buffer solution interface was the primary factor that determined the parabolic shape of the latex particles.

"5. The separation of the 0.2μ and 0.8μ latex particles in Tube 1 did in fact occur according to theory...." (1, p. 278)

Key Words: Biotechnology, Electrophoresis, Aqueous Solutions, Separation of Components, Electric Field, Electrodes, Monodisperse Latex Particles, Suspension of Particles, Particle Dispersion, Particle Migration, Particle Mobility, Surface Charge, Convection, Thermal Convection, Sedimentation, Medical Applications, Electroosmosis, Wall Effect, Particle Motion, Bubbles, Solid/Liquid Interface, Liquid/Liquid Interface, Contained Fluids

Number of Samples: three
Sample Materials: First tube: a mixture of 0.2 and 0.8 micron diameter particles; second tube: 0.8 micron diameter particles; third tube: 0.2 micron diameter particles.
Buffer: dilute borate (H_3BO_3/NaNOH)
(H*B*O*Na*N*O*H*)
Container Materials: polycarbonate (Lexan^{TM}) tubes

Experiment/Material Applications:
The separation of cells by electrophoresis is an attractive technique because there is no permanent damage of the cells during processing. In the low-gravity environment, convective and sedimentary phenomena within the fluid system are reduced. Particles or molecules in the solution remain suspended (with minimal convective currents and settling of higher density cells).

Polystyrene particles employed in this experiment were used to simulate the separation of large biological particles.

References/Applicable Publications:
(1) Snyder, R. S., Bier, M., Griffin, R. N., Johnson, A. J., Leidheiser, H., Micale, F. J., Ross, S., Vanderhoff, J. W., and Van Oss, C. J.: Free Fluid Particle Electrophoresis on Apollo 16. Separation and Purification Methods, Vol. 2, 1973, pp. 259-282. (post-flight)

(2) Micale, F. J., Vanderhoff, J. W., and Snyder, R. S.: Analysis of Electrophoresis on Apollo 16. Separation and Purification Methods, Vol. 6, 1977, pp. 361-372.

(3) Snyder, R. S.: Electrophoresis Demonstration on Apollo 16. NASA TM X-64724, November 1972.

(4) Griffin, R. N. and McCreight, L. R.: Convectionless Electrophoretic Separation of Biological Preparations. Final Report, Contract NAS8-27797, 1972. (experiment setup)

(5) Electrophoresis Demonstration. In Summaries of Early Materials Processing in Space Experiments, Edited by R. J. Naumann and E. Darby Mason, NASA TM-78240, August 1979, p. 11. (post-flight)

(6) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(7) Input received from Principal Investigator R. S. Snyder, August 1993.

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Principal Investigator(s): Snyder, R. S. (1), Morrison, D. R., Sr. (2)
Co-Investigator(s): Todd, P. (3), Barlow, G. (4), Lewis, M. L. (5), Rhodes, P. H. (6)
Affiliation(s): (1,6) National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (2) NASA Johnson Space Center (JSC), Houston, Texas; (3) During STS-003: Pennsylvania State University, University Park, Pennsylvania, Currently: University of Colorado, Department of Chemical Engineering, Boulder, Colorado; (4) During STS-003: Michael Reese Foundation, Chicago, Illinois, Currently: Deceased; (5) During STS-003: NASA Johnson Space Center (JSC), Houston, Texas, Currently: University of Alabama in Huntsville

Experiment Origin: USA
Mission: STS Launch #3, STS-003 (STS OFT-3, Columbia)
Launch Date/Expt. Date: March 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Reflight of Apollo-Soyuz Test Project (ASTP) hardware with slight modifications
Builder of Processing Facility: Originally: NASA, Marshall Space Flight Center (MSFC), Huntsville, Alabama
Refurbishment: NASA MSFC; NASA JSC fabricated new electrophoresis glass columns

Experiment:
Electrophoresis Experiment Verification Test (EEVT) (Static Column Electrophoresis)

Biological cells, in the presence of an electric field, can be separated according to their surface electric charge. On Earth, such separation is hampered by gravitational affects. For example, heat produced by the electric field can initiate and sustain thermal convection in the fluid system. Further, cells of highest density settle to the bottom of system before separation can occur. Both of these gravity-induced phenomena result in a mixing of the sample within the buffer fluid. Thus, the separation of the cells on the basis of their electrical charge is defeated. In a low-gravity environment, such convective and sedimentary effects are greatly reduced and improved separation of target cells should be possible.

This STS-003 experiment was the second in a series of experiments designed by Snyder et al. to study reduced gravity electrophoresis (see Snyder, Apollo 16). Electrophoresis hardware, previously employed on the Apollo-Soyuz Test Project (ASTP) mission (see Allen, ASTP (this chapter) was reused on the

STS-003 flight to (1) repeat the most promising aspects of the original ASTP investigations and (2) determine "...the limiting concentration of cells that can be separated by free fluid zone electrophoresis." (2, p. 4)

The experimental setup consisted of "...an electrophoresis processing unit with [coated] glass columns in which the separation takes place; a camera and film to document the process, and a cryogenic freezer to freeze and store the samples after separation." (1, p. 64)

During the shuttle mission, cell samples, which were inserted into the electrophoresis unit by a crew member, were allowed to separate for 1 hour. After separation, the fluid columns were frozen to preserve the samples for post-flight analysis. A total of eight columns were processed: two containing fixed red blood cells (RBC) and six containing human kidney cells.

"The astronauts did not report any difficulties in conducting the experiments, and their descriptions of the RBC migrations yielded the predicted displacements of the leading edges of the bands. All columns were recovered and returned to JSC where column slicing and analyses were to have been done. The intent was to slice the frozen columns using procedures developed during ASTP, and then to measure and compare the significant particle and fluid properties within narrow slices of both the low and high concentration columns." (2, p. 6)

It was reported that "Because the columns were not maintained in a frozen state after return to Earth, it was not possible to obtain post-flight information on final distribution of cells, electrophoretic properties of cell fractions or properties of the buffer after electrophoresis." (2, p. 3) "The [STS-003] photographs of the [two] RBC migrations became the only source of data from the space experiments." (2, p. 6)

In the first (RBC) column, a high concentration of cells were to be separated. "As the sample of RBC first became visible at the left end of the column, there appeared to be a bubble of gas occupying the nose of the sample mass... the sequence of photographs shows a broad band of cells moving along the column with no apparant [sic] separation. This could be due to electroosmosis, concentration effects or distortion of the electric field by the bubble..." (2, p. 6)

In the second (RBC) column a low concentration of cells was examined. "The observed band structure was narrow, and no clear trailing band was seen. Densitometry measurements of the flight negatives indicated that a trailing band could have been present... but the densitometric evidence is not conclusive due

to the distribution of glare and shadows photographed in the columns." (2, p. 6)

It was concluded that despite (1) post-flight procedural anomalies, (2) a bubble preceding the leading edge of the high concentration RBC column, and (3) a questionable trailing band in the low concentration RBC column, detailed analyses of RBC cell migration from photographs taken during STS-3 affirmed "...that particle electrophoresis in space is not perturbed by unexpected secondary effects attributable to the microgravity environment or to cell concentration." (2, p. 9)

Key Words: Biotechnology, Electrophoresis, Aqueous Solutions, Static Column Electrophoresis, Separation of Components, Particle Dispersion, Suspension of Particles, Particle Migration, Particle Distribution, Density Difference, Liquid Transfer, Liquid Injection, Liquid/Liquid Interface, Solid/Liquid Interface, Electric Field, Electrodes, Surface Charge, Electroosmosis, Wall Effect, Thermal Convection, Sedimentation, Bubbles, Coated Surfaces, Biological Cells, Model Systems, Contained Fluids, Cell Preservation, Freezing, Cell Storage, Thermal Environment More Extreme Than Predicted, Photographic Difficulties, Illumination Difficulties, Medical Applications, Pharmaceutical Applications, Biological Materials Processing, Deterioration of Samples After Low-G Flight

Number of Samples: eight separating columns
Sample Materials: (1) live human kidney cells; (2) human and rabbit red blood cells. Suspending Buffer: 5% (v/v) dimethyl sulfoxide (DMSO)
Container Materials: glass columns

Experiment/Material Applications:
These electrophoresis investigations were performed on STS-003 to reduce convective and sedimentation effects during the separation of biological cells.

Fixed red blood cells are considered a good model system when testing the separation capabilities of an electrophoresis unit.

An actual pharmaceutical product could be obtained from the separation of kidney cells. Specifically, kidney cells which produce urokinase were targeted for separation from the rest of the kidney cells in the sample medium.

Because electrophoretic separation does not result in cell damage, target cells are used for a variety of endeavors including immunology studies and cell-biology research.

References/Applicable Publications:

(1) NASA STS-3 Third Space Shuttle Mission Press Kit, March 1982, pp. 64-65. (preflight)

(2) Snyder, R. S., Rhodes, P. H., Herren, B. J., Miller, T. Y., Seaman, G.V.F., Todd, P., Kunze, M. E., and Sarnoff, B. E.: Analysis of Free Zone Electrophoresis of Fixed Erythrocytes Performed in Microgravity. Electrophoresis, Vol. 6, 1985, pp. 3-9. (post-flight)

(3) Memorandum for Record. Preliminary Results of Low Level G Force Monitoring During Conduct of Electrophoresis Equipment Verification Testing on STS-3, Cletis R. Booher, Lyndon B. Johnson Space Center, SE3/5-82/88, May 18, 1982. (preflight)

(4) Morrison, D. R. and Lewis, M. L.: Electrophoresis Tests on STS-3 and Ground Control Experiments: A Basis for Future Biological Sample Selections. 33rd International Astronautical Federation Congress, Paris, France, September 26-October 2, 1982, IAF Paper No. 82-152, 11 pp. (post-flight)

(5) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(6) Input received from Principal Investigator R. S. Snyder, August 1993.

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Principal Investigator(s): McGee, K. (1)
Co-Investigator(s): Patterson, A. (Teacher/Sponsor) (2)
Affiliation(s): (1) During Skylab: South Garland High School, Garland, Texas, Currently: Stone and Webster Engineering Corporation, Houston, Texas/Celestech Corporation, Houston, Texas; (2) South Garland High School, Garland, Texas

Experiment Origin: USA
Mission: Skylab Program
Launch Date/Expt. Date: Not applicable. Although this investigation was originally chosen as a Skylab student experiment, the associated hardware was never built and Principal Investigator K. McGee assisted with an Apollo 16 experiment (see Snyder, Apollo 16 (this chapter).
Launched From: Not Applicable
Payload Type: Skylab Student Project, High School Student Experiment, Skylab Manned Environment
Processing Facility: Planned: four rectangular chambers with (1) transparent ports for viewing and photography, (2) heaters for temperature control, and (3) an electric field generator.
Builder of Processing Facility: Not Applicable

Experiment:
The Effect of Zero Gravity on the Colloidal State of Matter

A substance with a supension of submicroscopic particles is called a colloidal system. The dispersed phase is surrounded by a dispersion medium or external phase (solid, liquid, or gas). Since there are three phases of matter, there are nine possible types of dispersions. For example, milk is a colloidal suspension of fat, protein, lactose, minerals, and vitamins in water (a liquid dispersed phase within a liquid dispersion medium). More importantly, the human body is primarily composed of colloidal matter; substances such as digestive fluids, body fluids, and blood are colloidal systems.

This Skylab student experiment was designed to study several effects of low gravity on colloidal chemistry. Four investigations in colloidal chemistry were proposed: (1) the formation of sol from two solutions, (2) the formation of a gel from two solutions, (3) the formation of a suspension (dispersion) by the addition of electrolytes to a solution (which cause the coagulation of the dispersed phase), and (4) the formation of several suspensions in a single tube by using electrodes placed in contact with the suspension (electrophoresis). These investigations were to be performed in an apparatus consisting of (1) four rectangular chambers with transparent ports for viewing and photography, (2) heaters for temperature control, (3) a power supply for electric field generation, and (4) a control system.

This experiment was initially selected as a feasible investigation for the Skylab student experiment program. However, in May 1971, "It was determined during Preliminary Design Reviews at the Marshall Space Flight Center... that the experiment as originally proposed would be extremely difficult, if not impossible, to perform as a Skylab Student Project experiment because of time, weight, and volume constraints." (3, p. 10) Therefore, the experiment was not performed during the Skylab program. Because the proposed experiment included electrophoresis, the student became involved with an Apollo 16 electrophoresis demonstration (see Snyder, Apollo 16 (this chapter)).

Key Words: Biotechnology, Colloidal Chemistry, Electrophoresis, Electric Field, Electrodes, Electrolytes, Gels, Dispersion, Particle Dispersion, Particle Distribution, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Solid/Liquid Dispersion, Solid/Liquid Interface, Stability of Dispersions, Stability of Suspensions, Suspension of Particles, Sedimentation, Coagulation, Contained Fluids, Medical Applications, Sample Not Processed As Planned

Number of Samples: unknown
Sample Materials: alloys, paper, paint, milk of magnesia, ammonia, jellies, glue, milk, protoplasm, steam, rubber, plaster, whipped cream, mayonnaise
Container Materials: unknown

Experiment/Material Applications:
"Colloidal theory can be utilized in many areas where the chemistry of regular solutions is insufficient in explaining phenomena. For example, colloidal chemistry is used to explain many structures in mineral ore, the colloidal nature of soil is important, and investigation of colloidal properties in metals and alloys has direct importance to space manufacturing.... Foods, for example gelatin, which is a liquid in solid colloid, exhibit colloidal properties.... More importantly...are the uses of colloidal chemistry in biological sciences." (3, p. 4)

References/Applicable Publications:

(1) "Student Investigations." In MSFC Skylab Mission Report, Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-71. (short description about student project)

(2) Skylab, Classroom in Space. Edited by Summerlin, L. B., NASA SP-410, 1977, p. 89. (post-flight of Skylab mission)

(3) McGee, K.: (report describing) The Effect of Zero Gravity on the Colloidal State of Matter. Author's Personal Publication, 30 pp.

(4) Input received from Principal Investigator K. McGee, August 1988.

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Principal Investigator(s): Bier, M. (1)
Co-Investigator(s): Snyder, R. S. (2), Wall, S. B. (3)
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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: November 1973-February 1974 (Mission Duration)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: Charged Particle Mobility Device (CPMD): A canister containing an assembly of two isotachophoresis (ITP) Plexiglas™ cells. The cells contained a buffer system suitable for ITP and a mechanism for insertion of samples while an electric field was applied.
Builder of Processing Facility: Construction of canister and electronic components: Astronautics Laboratory, NASA Marshall Space Flight Center (MSFC), Huntsville, Alabama.
Construction of the two ITP cells: Principal Investigator/Veterans Administration Hospital, Tucson, Arizona.

Experiment:
Role of Gravity in Preparative Electrophoresis
(Charged Particle Mobility TV117-SD35)

Isotachophoresis (ITP) is a variant of electrophoresis. In ITP, the test sample to be fractioned is inserted between a leader and terminator buffer. The leader buffer has a higher ion mobility than the sample and the terminator buffer has a lower ion mobility than the sample. As voltage is applied, sample components separate according to their electrophoretic mobility and form migrating self-sharpening zones. It is characteristic of ITP that the zones contain proteins or other biological materials in high concentration, establishing steep density gradients. Thus, on Earth, conventional ITP is often carried out in gels to prevent convective currents from occurring in adjacent zones.

This Skylab SL-4 experiment was the first in a series of investigations designed by Bier et al. to study electrophoretic-related separation of biological materials. The experiment was quickly prepared when the opportunity arose to include a simple electrophoresis experiment on the last Skylab mission.

Reportedly, the experiment was subject to three major limitations: (1) the small amount of time to prepare the experiment, (2) the limited power available to the experiment (28 volts) and (3) the "...requirement for the whole package to fit a volume defined by a cylinder of 3.5" diameter, 3.5" long." (1, p. 740)

The primary objectives of the research were to (1) establish if living red blood cells would form ITP zones in the absence of gravity, and (2) compare the resulting low-gravity resolution of two proteins in free solution with the resulting terrestrial resolution of two proteins in gel medium.

The Skylab experiments were conducted in cylindrical Plexiglas™ test cells. At one end of each cell, the leader buffer and leader electrode were housed. At the other end of each cell, a terminator buffer and a smaller terminal electrode were housed. The two buffers were separated by a movable partition which also contained the test specimen. Once the appropriate voltage was applied, the manually operated partition was removed and the sample advanced into the leader buffer. Component separation due to the electric field was monitored via an observation window in the cell. Two such experimental cells were employed: the first to examine the fractioning of two colored proteins (horse spleen ferritin and human hemoglobin) and the second to examine the fractioning of human blood cells.

During the space experiment, unexpected air bubbles were observed in both of the experiment cells. It was estimated that these bubbles were a result of fluid leakage. Nevertheless, forward migration was observed in the second cell (human red blood cells): "...the advancing front showed a little bowing, the view being partially obstructed by air bubbles... Upon completion of forward migration, the current polarity was reversed, and the astronauts then repeated the frontal migration a second time." (1, p. 741) During the second run, a very sharp profile was observed, although it was not known if the profile was due to electroosmosis alone. It was also noted that mixing of the buffers may have taken place during the second run.

Reportedly, after the partition was removed in the first cell and the colored proteins released, no observable migration was noted. Post-flight investigation indicated that the appropriate current was not produced in the cell. It was thought that the unexpected air bubbles may have hindered the current flow.

It was concluded that while the experiment was somewhat flawed by the procedures used during Skylab, it did prove the feasibility of ITP.

Key Words: Biotechnology, Isotachophoresis, Electrophoresis, Aqueous Solutions, Electric Field, Electrodes, Surface Charge, Particle Dispersion, Particle Mobility, Particle Migration, Separation of Components, Suspension of Particles, Density Difference, Convection, Sedimentation, Bubbles, Liquid Transfer, Liquid Injection, Liquid Mixing, Liquid Leakage, Biological Cells, Electroosmosis, Wall Effect, Contained Fluids, Liquid/Liquid Interface, Solid/Liquid Interface, Biological Materials Processing, Medical Applications, Processing Difficulties

Number of Samples: two
Sample Materials: First ITP cell- biological medium: a mixture of two colored proteins (horse spleen ferritin and human hemoglobin), leader buffer: 20 mM chloride-tris buffer, terminator buffer: 380 mM tris-glycine; Second ITP cell- biological medium: human red blood cells, leader buffer: 20 Mm chloride-tris buffer with 5% dextrose added for isotonicity. <Note: the terminator buffer was not specified.>
Container Materials: Plexiglas™

Experiment/Material Applications:
Reportedly, separation of cells in a low-gravity environment was and still is a major objective of the NASA space program. The experiment was designed to promote the concept of isotachophoretic separation in space, as the method provides the advantages of high resolution and high concentration.

"Because of the longtime delay between launch and performance of the experiment, necessitating an optional medium for cell survival... [one of the ITP cells] consisted of whole blood, with gentamycin as preservative." (8, p. 676)

The horse spleen ferritin/human hemoglobin mixture was chosen because "Extensive terrestrial trials had been conducted on this mixture showing that it separated well on gels in the presence of Ampholine." (8, p. 676)

References/Applicable Publications:
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(3) Naumann, R. J. and Herring, H. W.: Materials Processing in Space - Early Experiments. NASA SP-443, p. 86, 1980.

(4) Charged Particle Mobility. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-51 - 7-56.

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(11) Input received from Principal Investigator M. Bier, July 1989 and July 1993.

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Experiment Origin: USA
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Locker Experiment
Processing Facility: Isoelectric Focusing (IEF) Apparatus: A 65-pound experiment consisting of eight focusing glass columns, means of applying an electric field, and a camera to photograph the migration of colored proteins.
Builder of Processing Facility: Glass columns: Center for Separation Science, University of Arizona, Tucson, Arizona.
The remainder of the apparatus: NASA Marshall Space Flight Center (MSFC), Huntsville, Alabama.

Experiment:
Isoelectric Focusing Experiment (IEF)

Isoelectric focusing (IEF) is a powerful electrophoretic technique. During the process, biological materials are separated into components of high purity and resolution.

The method fractions samples in solutions according to their surface electrical charge. (The pH of the solution dictates the surface charge.) In the presence of an electric field, passage of a component through a pH gradient (toward the pole of opposite charge) effectively changes the component's charge. When the component passes into the appropriate pH zone, it has a surface charge of zero (its isoelectric point). Here the mobility is zero, and the constituent remains focused. On Earth, both gravity-dependent and gravity-independent factors drive fluid instabilities in the focusing process.

"One problem that develops in a simple glass wall tube configuration is electroosmosis in which the focused...[sample] is distorted by undesired flow patterns developed in the closed tube by the electric field. This is caused by the surface of the glass tube taking on a negative charge and inducing a positive charge in the fluid. The presence of the electric field then acts on the charged fluid to cause the outer layers to flow toward the negative electrode, and the center to flow toward the positive electrode." (6)

This STS-011 experiment was the second in a series of investigations designed by Bier et al. to study electrophoretic-related separation of biological materials (see Bier, Skylab, SL-4). The experiment was specifically designed to (a) minimize the gravity-dependent factors and (b) highlight the gravity-independent electroosmosis effects caused by surface electric charges.

Eight glass columns were employed. Buffer solutions forming different pH gradients were housed in the columns. <Note: a document published prior to the launch of the STS-011 mission (Reference (2)) indicated that the columns were to be coated to minimize electroosmosis.> During the space experiment, the migration of colored hemoglobin and colored albumin (in the presence of an electric field) was documented by a 35 mm camera. <Note: details of the specific contents of each of the columns were not presented in the available documents.>

Results (recorded in 40 photographs) indicated that isoelectric focusing in low gravity can result in the sudden onset of rapid fluid convection. This convection could not be readily ascribable to either gravity or electroosmosis effects. Subsequent ground-based work at NASA MSFC and the Center for Separation Science indicated that this fluid instability was most likely caused by electrohydrodynamics-a phenomenon not previously recognized in aqueous media. Plans were formulated to confirm this conclusion in subsequent experiments (see Bier, STS-26).

<Note: Reference (1) was not available to aid in the preparation of this experiment summary. Reference (7), which was provided by the Principal Investigator after this summary was prepared for publication, includes specific details of the IEF columns and experiment procedures.>

Key Words: Biotechnology, Isoelectric Focusing, Electrophoresis, Separation of Components, Electric Field, Electrodes, Surface Charge, pH Change, Aqueous Solutions, Suspension of Particles, Particle Dispersion, Particle Motion, Particle Migration, Particle Mobility, Biological Cells, Convection, Thermal Gradient, Electroosmosis, Wall Effect, Electrohydrodynamics, Hydrodynamics, Fluid Stability, Coated Surfaces, Contained Fluids, Solid/Liquid Interface, Liquid/Liquid Interface, Biological Materials Processing, Medical Applications

Number of Samples: eight
Sample Materials: two colored proteins, hemoglobin and albumin, in buffered solutions forming a pH gradient
Container Materials: glass

Experiment/Material Applications:
The experiment has applications for both materials processing and hydrodynamics. The low-gravity experiment, coupled with parallel ground research, resulted in the development of two new instruments for free-fluid focusing. Both of these instruments are now in commercial production. One of the instruments, aptly named Rotofor, actually simulates microgravity through rotation about its horizontal axis.

"Commercially produced mixtures of ampholytes are used because of their availability and cost. The biological samples used have a variety of components with discrete isoelectric points that allow well-defined separations." (2, p. 2)

References/Applicable Publications:
(1) Bier, M., Twitty, G. E., and Eagan, N. B.: Electroosmosis in Isoelectric Focusing-Ground Based and Space Experiments. Electrophoresis '84.

(2) Isoelectric Focusing (IEF): A Space Shuttle Middeck Materials Processing Experiment. Application Payload Projects, Spacelab Payload Projects Office, Marshall Space Flight Center, 5 pp. (preflight, prepared by Teledyne Brown Engineering)

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Experiment Origin: USA
Mission: STS Launch #26, STS-26 (Discovery)
Launch Date/Expt. Date: September 1988
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Isoelectric Focusing (IEF) Apparatus. The processing facility employed during the STS 41-B IEF experiment was reused (see Bier, STS 41-B), but some of the eight focusing glass columns were modified to have varying cross-sectional geometries.
Builder of Processing Facility: Glass columns: Center for Separation Science, University of Arizona, Tucson, Arizona.
The remainder of the apparatus: NASA Marshall Space Flight Center (MSFC), Huntsville, Alabama.

Experiment:
Isoelectric Focusing Experiment (IEF)

This STS-26 experiment was the third in a series of investigations designed by Bier et al. to study electrophoretic-related separation of biological materials (see Bier, Skylab SL-4, STS-011).

During Bier's earlier STS-011 isoelectric focusing (IEF) experiment, a sudden onset of rapid fluid convection was observed. Subsequent ground-based work indicated that the fluid instability was most likely caused by electrohydrodynamics. The STS-26 experiment, therefore, was designed to (1) confirm that the convection was due to electrohydrodynamics and (2) allow differentiation of fluid disturbances caused by (a) gravity, (b) electroosmosis, and (c) electrohydrodynamics. (In ground-based experiments, gravity is dominant, suppressing much of the other two effects. Electroosmosis is a surface phenomenon, while electrohydrodynamics is a bulk phenomenon.)

Eight glass focusing columns were prepared for the STS-26 experiment. The columns were similar to those employed on STS-011, but some were modified and had varying cross-sectional geometries. (The surface/volume ratio was varied in the columns to better study sources of fluid disturbances.) Buffer solutions forming an appropriate pH gradient were housed in the columns. <Note: specific details of the column modifications, buffer solutions,

and employed pH gradients were not detailed.>

During the space experiment, an electric field was placed across the columns and the migration of two colored proteins (hemoglobin and albumin) was documented by a 35 mm camera.

Results were recorded in 40 photographs. It was concluded that the experiments confirmed the importance of electrohydrodynamics in aqueous fluid systems. Reportedly, this confirmation was of major theoretical and practical relevance.

Further detailed information concerning this experiment could be located at this time. <Note: Reference (4) could not be procured at this time. Reference (6), which was provided by the Principal Investigator after this summary was prepared for publication, includes further details of the IEF columns and experiment procedures.>

Key Words: Biotechnology, Isoelectric Focusing, Electrophoresis, Separation of Components, Electric Field, Electrodes, Surface Charge, pH Change, Aqueous Solutions, Suspension of Particles, Particle Dispersion, Particle Motion, Particle Migration, Biological Cells, Convection, Thermal Gradient, Fluid Stability, Electroosmosis, Wall Effect, Container Shape, Aspect Ratio, Electrohydrodynamics, Hydrodynamics, Contained Fluids, Solid/Liquid Interface, Liquid/Liquid Interface, Biological Materials Processing, Medical Applications

Number of Samples: eight
Sample Materials: Each of the eight glass cells contained two colored proteins (hemoglobin and albumin) in buffered solutions forming a pH gradient in an electric field.
Container Materials: glass

Experiment/Material Applications:
The experiment has applications for both materials processing and hydrodynamics. Free-fluid focusing is widely practiced in ground-based instruments for biological separations. The experiment contributed to the understanding of phenomenon observed in ground-based work (only at much higher electric potentials). Electrohydrodynamics is of interest to hydrodynamicists.

See also Bier, STS-011.

References/Applicable Publications:

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(2) Investigators Examining Every Particle of Data from STS-26 Experiments. In NASA Marshall Star Newspaper, Vol. 29, Number 16, November 16, 1988, NASA Marshall Space Flight Center, Huntsville, Alabama, p. 3. (post-flight)

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Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Manned Environment
Processing Facility: Electrophoresis Unit (EU) and a cryogenic freezer
Builder of Processing Facility: NASA Marshall Space Flight Center (MSFC), Huntsville, Alabama

Experiment:
Electrophoresis Technology (MA-011) (Static Column Electrophoresis)

"Most biological materials, when dissolved or suspended in a selected aqueous medium, have a characteristic electric charge, and the migration velocity per unit electric field strength (defined as the electrophoretic mobility of the material) is thus fixed.... If the mobility difference between biological species is small, separation can be enhanced by increasing the length of the [separation] column. Electrophoresis can be conducted in liquid media, or free solutions, but problems arise because of disturbances in the bulk of the fluid. Two major causes of these disturbances are sedimentation of the particles or solute being separated and thermal convection generated by Joule heating of the column during electrophoresis. Although various techniques have been developed to overcome these problems on Earth, the elimination of gravity-induced sedimentation and thermal convection can be accomplished best in the near-zero-g environment of space." (1, p. 308)

"Isotachophoresis is a relatively new technique of electrophoretic separation in which a discontinuous electrolyte system is used at the site of sample injection.... The boundaries between species of different mobility are sharply defined and stabilized by electrical forces. Adjacent compartments of sample components that may not be distinguishable by zone electrophoresis may be resolved by isotachophoresis. The boundaries are highly self-recuperative and will reform if stirred or disrupted by other factors, including convection. The concentration of each substance within a compartment is uniform and remains constant throughout the run once the separation has been achieved. Because of this uniformity, higher concentrations of components can be handled with no deterioration of the sharp boundaries." (1, p. 309)

The specific objectives of this Apollo-Soyuz Test Project (ASTP) experiment were to separate biological cells by (1) electrophoresis and (2) isotachophoresis.

Prior to the flight (a) eight, 6-inch long glass columns were filled with an appropriate buffer solution and (2) slides containing a frozen sample of either red blood cells, human lymphocytes, or human fetal kidney cells were prepared. Six of the columns (numbered 1,2,3,5,6, and 7) were prepared for zonal electrophoresis and two of the columns (numbered 4 and 8) were prepared for isotachophoresis. (See Sample Materials section for (1) a description of the buffer in each column and (2) the number of cells loaded on a sample slide.)

The ASTP Electrophoresis Unit (EU) was capable of processing one column at a time. During the mission, each of the glass columns was configured into the EU and the appropriate sample slide inserted into the column. Zonal examinations included: fixed rabbit, human, and horse red blood cells (columns 1 and 5), human lymphocytes (columns 2 and 6), and human fetal kidney cells (columns 3 and 7). Isotachophoresis examinations included fresh human and rabbit blood cells (column 4) and fixed rabbit and human red blood cells (column 8). A 70 mm camera documented each column separation. After separation, electrophoresis columns were frozen by a thermoelectric module and placed in a cryogenic freezer for return to Earth. Isotachophoresis columns were not frozen for post-flight analysis. <Note: It appears that column 7 was not subjected to electrophoresis in orbit as planned: "...crewmembers discontinued the processing of column 7." (1, p. 324)>

In the ground-based laboratory, frozen samples were sliced and analyzed to determine the number and type of separated cells. Reportedly, "A separation of the three types of fixed blood cells (rabbit, human, and horse) was demonstrated. The human lym-

phocytes, however, showed no apparent migration." (1, p. 307) In addition it was noted that separation of "...human kidney cells produced the most exciting data. Analysis shows electrophoretic separation throughout the entire column with at least four bands of viable cells." (1, p. 307)

Isotachophoretic separation was also achieved. Reportedly, "...the two isotachophoresis experiments did not run for a sufficient time to permit visualization of rear or intercompartmental boundaries. Nevertheless, the experiment shows the great advantage of isotachophoresis in the sharpness of the frontal boundary and in the concentration of the migrating zones. Thus, the main potential advantage of isotachophoresis is the quality of sample it may fractionate. With respect to cells, however, one must specify that the future of isotachophoresis depends on finding proper spacers." (1, p. 333)

Extensive information on each of the eight column separations can be found in Reference (1).

It was reported that the experiment proved that living cells could be transported into space, separated in the low-gravity environment, returned to Earth for analysis, and then grown in the laboratory. Further, the experiment helped "...open the door for all space processing of biological materials via separation science." (9)

Key Words: Biotechnology, Electrophoresis, Isotachophoresis, Static Column Electrophoresis, Aqueous Solutions, Electric Field, Electrodes, Electrolytes, Suspension of Particles, Particle Dispersion, Particle Mobility, Particle Migration, Sedimentation, Concentration Distribution, Surface Charge, Separation of Components, Fluid Stability, Liquid Injection, Thermal Convection, Coated Surfaces, Cell Preservation, Cell Storage, Freezing, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Biological Materials Processing, Biological Cells, Pharmaceutical Applications, Medical Applications, Blood Clotting, Incomplete Sample Processing

Number of Samples: eight
Sample Material: The buffer in all six electrophoresis columns was the same: a mixture of 1.76 mmol Na_2HPO_4, 0.367 mmol KH_2PO_4, 6.42 mmol NaCl, 0.336 mmol Na_2ethylenediaminetetraacetic acid, 222 mmol glucose and 514 mmol glycerol in water. It had a hydrogen-ion concentration (pH) of 7.30 +/- 0.10 at 293 K and a

calculated ionic strength of 0.0097 mol/liter. At 298 K, the conductivity was 0.96 mmho/cml, the density was 1.022, and the dynamic viscosity was 0.0011 N-sec/m^2.

The buffers in both isotachophoresis columns were the same: (1) a leader buffer of 0.62 ml of 85% phosphoric acid in 500 ml water, with 42 g of dextrose and 276 g of glycerol, adjusted to a pH of 7.4 in 1 liter of distilled water, and (2) a terminator buffer containing 2 g of serine, 42 g of dextrose, and 276 g of glycerol, adjusted to pH 8.2 in 1 liter of glass distilled water.

Zonal examinations included: fixed rabbit, human, and horse red blood cells (columns 1 and 5), human lymphocytes (columns 2 and 6), and human fetal kidney cells (columns 3 and 7). Isotachophoresis examinations included fresh human and rabbit blood cells (column 4) and fixed rabbit and human red blood cells (column 8).

The number of red blood cells loaded in the sample slide was 5.22 X 10^6 rabbit cells/0.06 ml, 3.44 X 10^6 human cells/0.06 ml, and 7.26 X 10^6 horse cells/0.06 ml. The number of human lymphocytes loaded in the sample slide were 1.5 X 10^7 lymphocytes/0.06 ml. The number of human kidney cells loaded onto a sample slide was 2.0 X 10^6.

Container Materials: It appears that all of the columns were transparent coated Pyrex™ tubes. All eight of the columns were approximately the same size: 0.38 inches outer diameter, 0.25 inner diameter, and 6 inches long.

Experiment/Material Applications:
Compared to terrestrial electrophoretic operations, low-gravity electrophoretic separations can be realized at lower voltages for longer periods of times. Thus, such low-gravity processing can lead to better cell separations with less cell damage.

"...the red blood cells used on the ASTP flight... provide an almost indestructible sample material resistant to mechanical stress, hemolytic agents, and surface modification. These fixed blood cells are stable for months under varying temperature conditions and extensive electrophoresis mobility measurements of the cells using a variety of buffers have been published.... Each type of cell was selected to be morphologically distinguishable under microscopic examination. The red color of the cells makes them clearly distinguishable on the photographs made in orbit." (1, p. 310)

"The interest in specific lymphocytes stems from the increased emphasis in the field of immunology...." (1, p. 310) (See Reference (1) for a complete discussion on the benefits of separating these materials.)

"The isolation and production of the enzyme urokinase (UK) has interested biological laboratories for more than 20 years... [as the] enzyme is capable of effecting the conversion of plasminogen to plasmin. This conversion is necessary to accomplish blood clot lysis.... UK has been isolated from cultures of cells located in the cortex of the kidney.... [O]nly approximately 5 percent of the cells in the cortex of the kidney produce UK. Obviously, if these 'producing cells' could be isolated and subjected to subculturing techniques a twenty-fold increase in the yield... might result. Electrophoresis has been used to try to isolate these 'producing cells'." (1, pp. 310-311)

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(2) Seaman, G.V.F., Allen, R. E., Barlow, G. H., and Bier, M.: Detailed Results of ASTP Experiment MA-011. In ESA SP-114, pp. 155-166. (post-flight)

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(5) Naumann, R. J. and Mason, E. D.: Electrophoresis Technology. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 50-51. (post-flight)

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(9) Input received from Principal Investigator R. E. Allen, July 1989.

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Experiment Origin: Federal Republic of Germany
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Manned Environment
Processing Facility: Electrophoresis experiment apparatus (separation chamber)
Builder of Processing Facility: Unknown

Experiment:
Electrophoresis (MA-014)

During free-flow electrophoresis, "...the sample to be separated is introduced continuously into a separation chamber, in which a buffer solution flows laminarly. An electric field is generated perpendicular to the direction of flow. Particles having different surface charge densities are deflected from the flow direction of the buffer by an angle determined by the flow rate and the electrophoretic mobility of the particles. After leaving the lower end of the separation chamber, the separated zones can be collected continuously or analyzed by evaluating the deflection and the density distributions of the fractions." (1, p. 336)

On Earth, continuous electrophoretic separation is limited by gravity-induced thermal convection and sedimentation. For example, application of current to the buffer medium produces small temperature gradients in the fluid and thermal convection results. To prevent such difficulties, the terrestrial-based separation system must be equipped with a very thin separation chamber. However, this thin chamber limits the throughput of liquid buffer and sample material and introduces unwanted wall effects on the fluid. It was suspected that in a space-based system (a) the throughput could be successfully increased and (b) the separation resolution could be improved.

This Apollo-Soyuz Test Project (ASTP) experiment was the first in a series of investigations designed by Hannig to study continuous flow electrophoresis under low-gravity conditions. The specific objective of the experiment was to investigate and evaluate the increase in sample flow and sample resolution in a space-based electrophoresis chamber.

<Note: A detailed description of the ASTP continuous flow electrophoresis system and its operation is presented in Reference (1), p. 340. The major aspects of the system are presented here.>

The continuous flow electrophoresis system had a flow chamber 28 mm wide and 3.8 mm thick. (These chamber dimensions are significantly larger than those allowed under 1-g conditions.) Separation occurred over 180 mm (the electrode length).

During the mission, four different samples were separated in the chamber: (1) rat bone marrow cells, (2) mixtures of human and rabbit erythrocytes, (3) rat spleen cells, and (4) rat lymph node cells (with the addition of human erythrocytes as markers).

"During the experiment sequence, the walls of the separation chamber were cooled down and the temperature was controlled at the operational condition of 278...[+/-] 3K.... The buffer leaves the storage container and enters the separation chamber somewhat upstream of the sample inlet to achieve a constant laminar flow in the separation chamber parallel to the electrodes. After having passed the separation chamber, the mixture of sample and chamber buffer entered the waste container.... The angle of deflection for a particular particle was determined by the separation buffer flow rate and the field gradient." (1, p. 340)

"The electrophoresis equipment was designed for the separation of [the] four mixtures of biological cells with variable sample flow rates, buffer flow rates, and electric field gradients." (1, p. 335) Four experimental steps for each sample were performed: (1) the apparatus was filled with the sample fluid such that any possible remainders of the previous sample were removed from the separation chamber, (2) initial settings were applied: 60 V/cm field strength, 0.275 cm^3/s buffer throughput, and 5 cm^3/hr sample flow rate, (3) the flow rate was decreased from 5 to 1 cm^3/hr while the field strength and buffer throughput remained the same (as in (2)), and (4) the field strength and buffer throughput were reduced to two-thirds their initial value with the sample flow rate kept at 1 ml/hr.

The separated biomaterial fractions were not collected. Instead, "The separation was evaluated by optoelectronic methods. By this method, a narrow slit across the downstream end of the separation chamber, parallel to the electric field is illuminated. The light passes through the quartz plates of the separation chamber and is attenuated as a function of the density of the biomaterial distribution. On the other side of the chamber, the light intensity, related to the cell density, is measured with a photo-diode array." (1, p. 340) The photovoltages of the diodes were succes-

sively scanned and digitally recorded on magnetic tape. Reportedly, the data collection rate was approximately 2.5 kilobits/sec with a brightness resolution of 1% per step.

After the ASTP mission it was reported that all systems of the continuous flow electrophoresis apparatus worked well except for the illumination system. "During development work, a halogen lamp was introduced into the system to save energy and it was not taken into account that the lack of gravity would cause a lack of thermal convection of the gasfilling <sic> of the lamp. This resulted in a higher temperature and therefore in greater lamp brilliance than under 1g conditions. Unfortunately the measuring range of the analog-to-digital converter was limited from 70 to 100% of transmission in order to use the full accuracy. As a consequence, the ADC was overranged by the high brilliance of the lamp and no normal absorption pattern could be found on the tapes." (4, p. 138)

"In the entire separation process, no 'true' cell distribution curve was obtained. The baseline of registration in all cases was above the maximum absorption obtained in the peaks. Because of this finding, an analysis of the experiment seemed impossible at first. However, during the stationary phase of the separation (after adjustments to constant experimental conditions), i.e., during 90-second separation time, irregularly occurring pulses (called 'events') were recorded. This pattern reflected the expected course of the separation curves...[see Reference (1) for these curves].... These pulses seemed to be caused by cell aggregation in the region of the separation bands." (1, p. 350)

To investigate the above assumption, ground tests were conducted and it was discovered that clusters of cells (5 to 10 in number) migrate along the same flow paths as single cells (see Reference (4) for complete explanation). Therefore, only the first experimental step was evaluated (the lack of sample throughput in the other experimental steps did not produce enough clusters for evaluation). Based on this evaluation, the following was reported:

(1) For rat bone marrow: Only 73 'events' were observed because these cells tend not to aggregate. However, the distribution pattern shows good separation.

(2) For rat spleen cells: A high quality separation occurred (358 events recorded). The results indicated a 1.5 times higher resolution at six-fold higher sample throughput for low-gravity electrophoresis.

(3) For human and rabbit erythrocyte mixture: Because these cells have a weak tendency to aggregate, no information could be ob-

tained.

(4) For rat lymph node cells: A high quality separation occurred (549 events recorded).

The following conclusions were among those reported:

(1) The applicability of free-flow electrophoresis under low-gravity conditions was confirmed.

(2) The possibility of separating living cells under low-gravity conditions was demonstrated.

(3) "A sample throughput greater than tenfold was achieved by the expansion of the separation chamber cross section, possible in space flight, and with other similar chamber dimensions." (1, p. 352)

(4) "Despite the error that occurred in the optical detection system, it was demonstrated that the separation sharpness corresponded at least to that of analytical separations under one-g conditions." (1, p. 352)

Key Words: Biotechnology, Electrophoresis, Continuous Flow Electrophoresis, Liquid Injection, Liquid Transfer, Aqueous Solutions, Electric Field, Electrodes, Separation of Components, Particle Dispersion, Suspension of Particles, Particle Mobility, Particle Migration, Particle Aggregation, Particle Distribution, Surface Charge, Biological Cells, Thermal Convection, Thermal Gradient, Sedimentation, Flow Rates, Wall Effect, Electroosmosis, Container Shape, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Halogen Lamps, Illumination Difficulties, Biological Materials Processing, Medical Applications, Thermal Environment More Extreme Than Predicted

Number of Samples: Four set of cells were separated.
Sample Materials: (1) rat bone marrow cells, (2) a mixture of human and rabbit erythrocytes, (3) rat spleen cells, and (4) rat lymph node cells (with the addition of human erythrocytes as markers). (A listing of buffer solutions is located in Reference (4).)
Container Materials: unclear

Experiment/Material Applications:
"To understand the specific function of cells and their membrane systems, research is needed concerning the problem of separation and characterization of particles performing individual functions. Of particular importance in studying cooperative interactions in biological processes is the isolation of the interacting systems in the most homogeneous and still functioning form. These problems are not only of a fundamental theoretical interest but are also of practical importance to elucidate normal and pathological processes in biology.

"The reason for using electrophoresis to investigate these problems concerns the properties of the cell membrane surface, which is the site of many important biological phenomena. Transformation and differentiation processes are often accompanied by changes of the cell surface charge density. Charge density, however, determines the electrophoretic behavior; i.e., the electrophoretic mobility. Therefore, electrophoresis is a highly efficient method of separating cells or other biological particles according to functional criteria. Furthermore, cell electrophoresis is one of the few physicochemical processes that can be applied to living cells without producing damage or loss of viability.

"Because electrophoresis is established as one of the most effective separation methods used in biological study, it is considered an essential tool for investigation in space. The expectation was that the separation capability would be increased at zero-g, resulting in new applications that depend on improved resolution." (1, pp. 335-336)

Reportedly, the specific cell mediums were chosen because each was a "known biological sample."

References/Applicable Publications:
(1) Hannig, K., Wirth, H., and Schoen, E.: Electrophoresis. In Apollo-Soyuz Test Project Summary Science Report, Vol. 1, NASA SP-412, 1977, pp. 335-352. (post-flight)

(2) Hannig, K. H., Wirth, H., and Schoen, E.: Electrophoresis Experiment MA-014. In Apollo-Soyuz Test Project--Composite of MSFC Final Science Report, Section II, NASA TM X-73360, January 1977, pp. II-1 - II-38. (post-flight)

(3) Naumann, R. J. and Mason, E. D.: Electrophoresis. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 52-53. (post-flight)

(4) Hannig, K. and Wirth, H.: Detailed Results of the ASTP Experiment MA-014. Material Science in Space, ESA SP-114, 1976, pp. 135-140. (post-flight)

(5) Hannig, K. and Wirth, H.: Free Flow Electrophoresis in Space. In Progress in Astronautics and Aeronautics, Vol. 52, 1977, pp. 411-422. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 18
Launch Date/Expt. Date: May 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-13 (free flow electrophoresis apparatus)
Builder of Processing Facility: Unknown

Experiment:
Electrophoretic Separation

This TEXUS 18 experiment was the second in a series of investigations designed by Hannig to study free flow electrophoresis under low-gravity conditions (see Hannig, ASTP).

Very little information concerning this experiment could be located in the available publications. The following brief summary was detailed in Reference (1).

"A new free flow electrophoresis apparatus was developed proceeding from the ACE 710, which had been designed in the department of Prof. Hannig between 1978 and 1982.... In collaboration with MBB-ERNO (Bremen, FRG) and Hirschmann Geraetebau (Unterhaching, FRG) the electrophoresis instrument was adapted for use under microgravity.... Its separation chamber has a length of 20 cm and a width of 7 cm. During the first test experiments the thickness of the buffer film was 0.5 mm. Under these conditions the machine could be operated at electrical field strengths up to 120 V/cm. The separated material is collected in 70 fractions.

"The machine was tested in the TEXUS flight of May 6, 1988 [TEXUS 18]. The aim of the test was to find out whether the new machine operates well under microgravity. For this purpose a mixture of rabbit, rat and guinea pig erythrocytes was electrophoresed.... [The results from the TEXUS 18 experiment]... demonstrated that the new electrophoresis machine is very suitable for cell electrophoresis under microgravity." (1, p. 92) <Note: The ACE 710 device was not further detailed in the reference.>

No other information concerning this experiment could be located.

Key Words: Biotechnology, Electrophoresis, Continuous Flow Electrophoresis, Liquid Injection, Liquid Transfer, Aqueous Solutions, Electric Field, Electrodes, Separation of Components, Particle Dispersion, Suspension of Particles, Particle Mobility, Particle Migration, Surface Charge, Biological Cells, Thermal Convection, Sedimentation, Flow Rates, Container Shape, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Biological Materials Processing, Medical Applications

Number of Samples: unknown, possibly one sample
Sample Material: mixture of rabbit, rat, and guinea pig erythrocytes
Container Materials: unknown

Experiment/Material Applications:
See Hannig, ASTP

The reasons why the sample materials were selected for this experiment were not detailed in the available publications.

References/Applicable Publications:
(1) Hannig, K. and Bauer, J.: Free Flow Electrophoresis in Space Shuttle Program (Biotex). In Advances in Space Research, Vol. 9, Number 11, 1989, pp. 91-96. (post-flight)

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Co-Investigator(s): Richman, D. W. (3)
Affiliation(s): (1-3) During STS-004: McDonnell Douglas Astronautics Company, St. Louis, Missouri; (1) Currently: McDonnell Douglas Aerospace, Center for Cell Research, Penn State University, State College, Pennsylvania; (2) Currently: Consultant, Merritt Island, Florida; (3) Currently: McDonnell Douglas Space Systems, Huntington Beach, California

Experiment Origin: USA
Mission: STS Launch #4, STS-004 (STS OFT-4, Columbia)
Launch Date/Expt. Date: June 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES)
Builder of Processing Facility: Designed by McDonnell Douglas Astronautics Company, St. Louis, Missouri. Samples developed in consultation with Ortho Pharmaceutical Division of Johnson and Johnson, Raritan, New Jersey

Experiment:
Continuous Flow Electrophoresis System (CFES)

Biological cells, in the presence of an electric field, can be separated according to their surface electrical charge. Such a process is known as electrophoresis. More specifically, in continuous flow electrophoresis (CFE), "...a stream of sample material is continuously injected into a long, rectangular chamber that is filled with a flowing buffer solution (called the carrier fluid). As the sample flows upward through the chamber, an electrical field is applied across the flow." (1, p. 140) "Because particles have different charges and sizes, they will move at different speeds away from one electrode toward another with the opposite charge." (1, p. 139) "This lateral movement splits the sample into separate particle streams which then exit through different outlet ports at the top of the chamber and are collected in the separate test tubes." (1, p. 140)

Unlike earlier space electrophoresis experiments (see for example, Allen, ASTP or Snyder, STS-003 (both in this chapter)), CFE processes larger quantities of biological materials in a continuous stream. Because it was expected that higher volumes and higher concentrations of samples could be processed in the low-gravity environment, CFE experiments were performed during this STS-004 mission.

The experiment was the first in a series of investigations designed by Lanham et al. and McDonnell Douglas Astronautics Co. (MDAC) to study CFE in the space environment. The experimental setup consisted of a cooled separation unit, an experiment control and monitoring unit, and a small refrigerator for sample storage. The separation unit was larger than its corresponding ground-based laboratory version, allowing for increased volume sample separation.

During the mission, a crewman placed "...the sample materials into the unit... [started] the flow of the carrier fluid, and put in collection cassettes.... The system then automatically injected samples into the chamber, starting the separation process. When the process was complete... [the crewman] removed the collection cassettes and placed them in the refrigerator module.

"[The crewman]... also took photographs of the process during various stages of the separation. These pictures show streams of samples that contain 463 times more protein than can be separated in a ground chamber under the same conditions....

"Analysis of the separated samples makes it clear just how dramatic the advantages of working in space are.... We had been concerned that the highly concentrated proteins used on the space tests might get in each others way and not separate effectively. But this did not happen; the degree [i.e. purity] of separation was the same for space and ground tests." (1, pp. 141-142)

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Aqueous Solutions, Particle Dispersion, Suspension of Particles, Electric Field, Electrodes, Surface Charge, Particle Mobility, Convection, Sedimentation, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Flow Rates, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Cell Storage, Cell Preservation, Refrigeration, Medical Applications, Biological Materials Processing

Number of Samples: Six samples were separated.
Sample Materials: Three samples were of rat and egg albumin in equal parts with total concentrations of 1%, 10%, and 25%. Three samples were of a proprietary culture media with solution concentrations of 10%, 20%, and 25%.

Container Materials: Samples entered the Lexan™ separation chamber through a thin-walled glass tube. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Individual samples were collected and stored in vinyl receptacles within the collection cassettes.

Experiment/Material Applications:
Terrestrial continuous flow electrophoresis experiments are hampered by a number of difficulties. For example, because "...most proteins are more dense than the carrier fluid, gravity causes the sample to collapse in a blob around the inlet port..." (1, p. 141) Thus, "...most samples must be highly diluted to flow properly." (1, p. 141) This dilution results in a lower concentration of the protein within the carrier available for separation. In addition, "...convection currents within the chamber cause the particle streams to waver back and forth. When this happens, the streams exit through each other's outlets, a result that degrades the purity of the separation. To overcome this problem, a very thin separation chamber is used.... Unfortunately, a thin chamber restricts the size of the sample inlet, and this, in turn, restricts the amount of the sample that can be injected and separated." (1, p. 141)

In contrast to these Earth-based difficulties which limit the amount of material separated, space electrophoresis should allow a much higher quantity of material separation. For example, the space concentration levels are "..limited only by the solubility of the proteins and the carrier fluid." (1, p. 141) In addition, space processing should result in the reduction of convective currents, thus allowing larger cross-sectional diameter chambers and sample inlets.

The mixtures of rat albumin and egg albumin separated during the the experiments were chosen "...because the two albumins are fairly close in mobility and because, in using this pure mixture..." (1, p. 142) the machine's separation capability could be demonstrated.

References/Applicable Publications:
(1) Richman, D. W.: Electrophoresis Operations In Space--A Promising New Era Of Business In Space. In Manufacturing in Space, Proceedings Of the Winter Annual Meeting of ASME, Boston, Massachusetts, November 13-18, 1983, pp. 139-142. (post-flight)

(2) Snyder, R. S., Rhodes, P. H., and Miller, T. Y.: Continuous Flow Electrophoresis System Experiments on Shuttle Flights STS-6 and STS-7. NASA TP-2778, October 1987. (includes short summary of STS-004 results)

(3) NASA STS-4 Fourth Space Shuttle Mission Press Kit, June 1982, pp. 55-58. (preflight)

(4) NASA Mission Report: STS-4 Mission Simulates Operational Flight-President Terms Success "Golden Spike" in Space. MR-004, NASA Johnson Space Center, 4 pp. (post-flight; very brief summary)

(5) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities, Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

(6) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(7) Weiss, R. A., Lanham, J. W., Richman, D. W., and Walker, C. D.: Investigation of the Free Flow Electrophoretic Process, Final Report. McDonnell Douglas Astronautics Company-St. Louis Division, May 1979, Report MDC E2000, Vol. I, Executive Summary, 22 pp. (preflight)

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(11) Richman, D. W.: System for Hydro-Dynamic Compensation for Electrophoresis Crescent Distortion. U.S. Patent No. 4,309,268 issued January 5, 1982.

(12) Richman, D. W. and Rose, A. L.: Electrophoresis Chamber. U.S. Patent No. 4,310,408 issued January 12, 1982.

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(15) Richman, D. W.: Electrophoresis Operations in Space for Pharmaceutical Processing. In Proceedings of IEEE National Telesystems Conference, 1983, p. 374.

(16) Snyder, R. S. and Rhodes, P. H.: Electrophoresis Experiments in Microgravity. In Proceedings of AIAA/IKI Microgravity Sciences Symposium, 1991, pp. 205-208.

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Co-Investigator(s): Richman, D. W. (4)
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Experiment Origin: USA
Mission: STS Launch #6, STS-006 (STS 31-B, Challenger)
Launch Date/Expt. Date: April 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment (located along the port side wall)
Processing Facility: Continuous Flow Electrophoresis System (CFES). The STS-004 hardware was modified to the Block 2 configuration by including a higher performance water cooling system utilizing the orbiter payload cooling loop to improve dissipation of heat produced by the separation process.
Builder of Processing Facility: Designed by McDonnell Douglas Astronautics Company, St. Louis, Missouri. MDAC samples developed in consultation with Ortho Pharmaceutical Division of Johnson and Johnson, Raritan, New Jersey

Experiment:
Continuous Flow Electrophoresis System (CFES)

This experiment was the second in a series of investigations designed by Lanham et al. and McDonnell Douglas Astronautics Co. (MDAC) to study continuous flow electrophoresis (CFE) in the space environment (see Lanham, STS-004).

Because earlier STS-004 experiments indicated that a much higher quantity of biological material could be separated in the shuttle environment without degrading sample purity, the primary (McDonnell Douglas) goal of this shuttle mission was to increase the purity of the separated samples. In order to achieve this goal, modification of the STS-004 CFES unit was necessary. This modification, which included the addition of a Pump Accumulator Module (located in one middeck locker), improved the cooling system of the CFES and allowed dissipation of up to 1-kW of heat (generated during the separation process) through the orbiter payload cooling loop. As a result of the heat reduction, a higher voltage could be placed across the separation chamber. This higher voltage, coupled with a longer time during which

sample materials were subjected to the electric field, resulted in the migration of particles to greater lateral distances.

Reportedly, "The internal dimensions of [the] space CFES...[Lexan™] separation (flow) chamber is 16 cm wide, 120 cm long, and 0.3 cm thick.... [The] cooling jacket covers each broad chamber face with one electrode in each cooling chamber positioned diagonally across from each other to provide the electric field across the length of the chamber. The platinum electrodes are in contact with the separation chamber via slots cut into the chamber plates and are covered with a proprietary, porous membrane. The circulation of cooled electrolyte, from the bottom (flow entrance) of the chamber to the top through a serpentine passageway, establishes a uniform lateral temperature gradient and removes the bubbles formed at each electrode. Rectangular struts used to form the coolant passage also provide some support to the thin separation chamber plates. The sample enters the chamber through a thin-wall glass tube (0.1 cm inside diameter) located 11 cm from the curtain buffer entrance of the chamber. The buffer and separated sample fractions exit at the top of the chamber through a collection array of 197 Tygon tubes (0.068 cm inside diameter, 0.078 cm outside diameter) which span the width of the chamber." (2, p. 2) (Further descriptions of the CFES apparatus and experimental operation are detailed under Lanham, STS-004.)

During this mission, the investigators achieved "...more than four times better resolution or purity [in separation of both a mixture of rat albumin and egg albumin, and with a tissue culture product] than is possible on Earth." (1, p. 142)

Under a NASA/McDonnell Douglas Joint Endeavor Agreement, NASA, Marshall Space Flight Center was able to submit two of their own samples for processing in the McDonnell Douglas CFES. Among the primary (NASA) goals of these experiments were to (1) validate sample concentration effects reported by McDonnell Douglas from STS-004 and (2) evaluate the continuous flow electrophoretic separation of a material of high concentration and compare it with ground-based CFE attempts.

NASA, being subject to the constraints of the McDonnell Douglas CFES system and the choice of fluid buffer (2 mM barbital), opted to have the following two samples processed: (1) a high concentration of hemoglobin alone (8.7%) and (2) a lower concentration mixture of polysaccharide (0.5%) and hemoglobin (1.9%). Reportedly, "The band behavior obtained from processing the single species was used to define the performance of the space instrument. The low concentration mixture..., although still higher than can be processed at high resolution on Earth, would permit a comparison of the separations achieved before flight in

the various laboratory units." (2, p. 4)

Photographs taken during processing of the high concentrations sample indicated extensive band spreading which was attributed to "...the mismatch of electrical conductivity between the sample and buffer." (2, abstract) Photographs taken during processing of the lower concentration hemoglobin-polysaccharide sample illustrated that "...the stream was more compact with little indication of spreading." (2, p. 5)

Post-flight analysis of the separated materials from the collection tubes indicated that the separation of the hemoglobin/polysaccharide mixture was not as distinct as that achieved during similar ground-based laboratory testing (see Reference (2), p. 5).

<Note: Not all of the references listed below were available to aid in the preparation of this experiment summary.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Aqueous Solutions, Particle Dispersion, Suspension of Particles, Particle Mobility, Particle Migration, Electric Field, Electrodes, Electrical Conductivity, Electrolytes, Surface Charge, Convection, Sedimentation, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Flow Rates, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Container Shape, Sample Purity, Bubble Formation, Porous Material, Membranes, Heat Transfer, Thermal Gradient, Cell Preservation, Cell Storage, Medical Applications, Biological Materials Processing, Processing Difficulties

Number of Samples: During the mission six samples were separated. NASA processed two samples and MDAC processed four samples.
Sample Materials: NASA samples: (1) a high concentration of hemoglobin and (2) a mixture of hemoglobin and a polysaccharide. MDAC samples: Two samples were of rat and egg albumin in equal parts with total concentrations of 25%. Two samples were of a proprietary tissue culture media with solution concentrations of 25%.
Container Materials: Samples selectively entered the Lexan™ separation chamber through either one or three thin-walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Individual samples were collected and stored in vinyl receptacles within the collection cassettes.

Experiment/Material Applications:
The reasons why the MDAC samples were selected can be found under Lanham, STS-004.

"Hemoglobin was selected as the primary [NASA] sample candidate based upon its availability in large quantities as a single, molecular species; its visibility for easy analysis; its utility as an electrophoresis standard in laboratories; its availability as variants with different electrophoretic mobilities, and its stability in the cyanmethoglobin form." (2, p. 2)

"The second [NASA] sample was one type of pneumococcal capsular polysaccharide (PCP)... PCP type 6 had a distinctly higher mobility than...[the hemoglobin] with minimal variation and, although not colored, could be detected in low concentrations immunologically." (2, p. 2)

References/Applicable Publications:
(1) Richman, D. W.: Electrophoresis Operations In Space--A Promising New Era Of Business In Space. In Manufacturing In Space, Proceedings Of the Winter Annual Meeting of ASME, Boston, Massachusetts, November 13-18, 1983, pp. 139-142. (post-flight)

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(12) Richman, D. W.: Electrophoresis Operations in Space for Pharmaceutical Processing; Permanent Presence--- Making It Work. American Astronautical Society, Science and Technology Series, Vol. 60, 1985, pp. 11-16.

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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES), Block 2
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES)

This experiment was the third in a series of investigations designed by Lanham et al. and McDonnell Douglas Astronautics Company (MDAC) to study continuous flow electrophoresis (CFE) in the space environment (see Lanham, STS-004, STS-006).

Very little information concerning the Lanham/McDonnell Douglas experiments could be located at this time. It was briefly reported that: "Lanham/McDonnell Douglas' objective during this mission was to improve their understanding of the separation characteristics (specifically obtaining a separated sample purity four times the best demonstrated on Earth) of a specific proprietary cell culture media product having commercial potential." (2, p. 52) The STS-007 experimental setup and procedure were similar to that of the earlier STS-006 mission (see Lanham, STS-006). (10)

Further detailed information concerning the experimental results of the Lanham/McDonnell Douglas experiments could not be located at this time.

Under a NASA/McDonnell Douglas Joint Endeavor Agreement, NASA Marshall Space Flight Center was able to submit two of their own samples for processing in the McDonnell Douglas CFES. Originally, the goals of the NASA STS-007 experiments were to

build on the NASA results observed on STS-006. However, "Because MDAC changed the curtain buffer on STS-7 from a barbital buffer (pH 8.3) to an propionate buffer (pH 5.2), it was not possible to perform a follow-up experiment using hemoglobin and polysaccharide as processed on STS-6. The change in pH would have resulted in both the hemoglobin and polysaccharide becoming positively charged and migrating toward the cathode. Polystyrene latex particles (PSL) were, therefore, chosen for separation on STS-7 since they are known to be negatively charged at pH 5.2 and are produced in a range of sizes with different surface charge groups and surface charge densities." (1, p. 2) The primary (NASA) goal for this mission was, therefore, "...to evaluate the influence of the electrical properties of the sample constituents on the resolution of the continuous flow electrophoresis device." (2, abstract)

Three PSL particle sizes were chosen. Those with the highest mobility were dyed red, those with the lowest mobility were dyed blue, while those with an intermediate mobility were not dyed. For both of the NASA samples, volumes of each particle size were combined to yield equal concentrations. However, the conductivity of the first sample to be processed was three times that of the second sample to be processed. Descriptions of the CFES apparatus and experimental operation are detailed under Lanham, STS-004 and Lanham, STS-006.

Post-flight, collected fractions of the samples and photographs of the separation were analyzed. "As expected, the polystyrene latex microspheres dispersed in solution with three times the electrical conductivity of the curtain buffer[,] separated with significantly larger band spread than in the second experiment under matched conductivity conditions." (1, abstract)

Further detailed information concerning the experimental results of the NASA experiments could not be located at this time.

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Electrical Conductivity, Separation of Components, Biological Cells, Aqueous Solutions, Monodisperse Latex Particles, Polystyrene Latex Microspheres, Particle Size Distribution, Particle Dispersion, Suspension of Particles, Particle Mobility, Electric Field, Electrodes, Surface Charge, Convection, Sedimentation, Liquid Expulsion Through a Small Orifice, Liquid

Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Sample Purity, Contained Fluids, Cell Storage, Cell Preservation, Medical Applications, Pharmaceutical Applications, Biological Materials Processing

Number of Samples: During the mission six samples were separated. NASA processed two samples and MDAC processed four samples.
Sample Materials: NASA: (1) a mixture of three different sizes of polystyrene latex particles with a total latex concentration of 5.0% and a conductivity of 155 +/- 5 micro-mhos/cm and (2) a mixture of three different sizes of polystyrene latex particles with a total latex concentration of 5% and a conductivity of 455 +/- 5 micro-mhos/cm.
McDonnell Douglas: The four samples were of proprietary tissue culture media with solution concentrations of 7.3%, 10%, and 25% (two samples)
Container Materials: Samples selectively entered the Lexan™ separation chamber through either one or three thin-walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Individual samples were collected and stored in vinyl receptacles within the collection cassettes.

Experiment/Material Applications:
The specific reasons why the PSL particles were chosen are stated in the experiment summary (above).

If convective effects are sufficiently suppressed during low-gravity CFES experiments, improved separation of biological materials might be realized in a high volume, high-quality pharmaceutical refining device.

See also Lanham, STS-004.

References/Applicable Publications:
(1) Snyder, R. S., Rhodes, P. H., and Miller, T. Y.: Continuous Flow Electrophoresis System Experiments on Shuttle Flights STS-6 and STS-7. NASA TP-2778, October 1987. (post-flight)

(2) NASA STS-7 Seventh Space Shuttle Mission Press Kit, June, 1983, pp. 52-54. (preflight)

(3) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities, Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

(4) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

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(10) Personal Communication with C. D. Walker (McDonnell Douglas Aerospace-Space Systems), August 1993.

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Experiment Origin: USA
Mission: STS Launch #12, STS-014 (STS 41-D, Discovery)
Launch Date/Expt. Date: August 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES) modified after STS-008 to the Block 3 configuration with changes to provide continuous single, large volume sample delivery and discrete collection in an isolated reservoir in lieu of several small samples. The configuration change also included modified cooling system components and the addition of a carrier buffer recirculation and filtration loop.
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES)

This space shuttle experiment was the fifth in a series of investigations designed by Lanham and McDonnell Douglas Astronautics Company (MDAC) to study Continuous Flow Electrophoresis (CFE) in the space environment (see Lanham, STS-004, STS-006, STS-007; Scharp, Morrison and Hymer, STS-008 (all in this chapter)). The specific objectives of the experiment were to (1) separate a large enough quantity of a specific hormone protein such that sufficient clinical testing and research of the material would lead to a new pharmaceutical product and (2) "evaluate the production system in concept". (7)

During the mission, a continuous stream of the concentrated protein material was injected into a buffer solution in the MDAC Continuous Flow Electrophoresis System (CFES). An electric field, placed across the fluid chamber within CFES, resulted in the separation of the protein solution into distinct streams according to their surface electrical charge.

The CFES had been modified since STS-008 to now operate for approximately 100 hours and to separate and collect one large

sample rather than several small samples. Because of the extended operating time and unique experiment procedures, McDonnell Douglas engineer Charles Walker attended to the experiment as Payload Specialist during the flight (the first non-career astronaut to operate a customer payload).

Reportedly, "When an anomaly curtailed operation on the Continuous Flow Electrophoresis System (CFES) experiment, payload specialist Charles Walker made the necessary workaround and changeout of equipment. The CFES had several shutdowns but accomplisned 85 percent of its planned objectives. (Later it was revealed that the experiment had been contaminated and lost its scientific value). The CFES demonstration, which was just at the brink of showing practicality, will be flown again on a future mission." (2) (For a summary of the results from the "future mission" see Lanham, STS-023.)

A more detailed discussion of the observed contamination was provided in Reference (4): "...post flight assays revealed levels of endotoxin contamination which which rendered the hormone unsuitable for animal testing." (4, p. 14)

Further information concerning the objectives, setup and results of this experiment could not be located at this time.

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Particle Dispersion, Particle Mobility, Electric Field, Surface Charge, Convection, Sedimentation, Medical Applications, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Contamination Source, Pharmaceutical Applications, Hardware Malfunction, Processing Difficulties

Number of Samples: one
Sample Materials: concentrated cell culture fluid containing the protein erythropoietin
Container Materials: Samples entered the Lexan™ separation chamber through two thin-walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Selected individual samples were collected and stored within a cooled polypropylene reservoir.

Experiment/Material Applications:
"McDonnell Douglas will separate material in increasing quantities so that Ortho [Pharmaceutical Corporation, Raritan, N. J.], will be able to conduct research and clinical testing needed to gain Food and Drug Administration approval for a new pharmaceutical product." (1, p. 20)

References/Applicable Publications:
(1) NASA Space Shuttle Mission 41-D Press Kit, June 1984, pp. 20-22. (preflight)

(2) Reporter's Space Flight Note Pad. Rockwell International Space Transportation Systems Division, Office of Public Relations, August 1985. (STS-014 mission facts; no page number listed; post-flight)

(3) Debe, M. K.: Industrial Materials Processing Experiments Onboard the Space Shuttle Orbiter. J. Vac. Sci. Technol. A, 4(3), 273, 1986. (identifies biological material processed)

(4) NASA Space Shuttle Mission 51-D Press Kit, April 1985, p. 14. (post-flight mission STS-014; discusses some STS-014 results)

(5) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities, Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

(6) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(7) Input Received from Investigators J. W. Lanham, D. W. Richman, and C. D. Walker, August 1993.

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(9) Richman, D. W. and Rose, A. L.: Electrophoresis Chamber. U.S. Patent No. 4,310,408 issued January 12, 1982.

(10) Richman, D. W.: System for Hydrodynamic Compensation for Electrophoresis Crescent Distortion. U.S. Patent No. 4,383,905 issued May 1983.

(11) Richman, D. W. and Walker, C. D.: Electrophoresis Apparatus with Flow Control. U.S. Patent No. 4,394,246 issued July 19, 1983.

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Experiment Origin: USA
Mission: STS Launch #16, STS-023 (STS 51-D, Discovery)
Launch Date/Expt. Date: April 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES), Block 3
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES)

This experiment was the sixth in a series of investigations designed by Lanham et al. and McDonnell Douglas Astronautics Company (MDAC) to study Continuous Flow Electrophoresis (CFE) in the space environment (see Lanham, STS-004, STS-006, STS-007, STS-014; Scharp, Morrison, and Hymer, STS-008 (all in this chapter)). The specific objectives of the experiment included (1) the separation and collection of a protein sample and (2) the study of the sample stream dynamics. Reportedly, "McDonnell Douglas expects to process 1.1 liters of concentrated protein material over the course of 3 flight days. On the final flight day, nine separate tests will be conducted to determine the optimum ratio between sample and buffer concentrations." (1, p. 14)

Because fluid contamination occurred on the earlier STS-014 mission, an additional objective of the experiment was to evaluate apparatus/sample contamination control.

Prior to the shuttle launch, the MDAC Continuous Flow Electrophoresis System (CFES) unit was cleansed with stronger sterilizing chemicals than had been previously employed. In flight, a cooler operating temperature of the CFES unit was prescribed to retard bacterial growth. In addition, payload specialist Charlie Walker tested daily for the presence of

microbes and endotoxins by "...withdrawing a small sample of fluid from five locations and incubating them in vials which have been been loaded previously with freeze-dried reactants." (1, p. 14; also Reference (10))

"The second objective of the mission was addressed with the injection of eight sample solutions. Seven were specially prepared to provide data on the concentration, conductivity and viscosity interactions of sample and buffer fluid within the CFES. The eighth used a small residual volume of the primary sample material to characterize electroosmotic effects on sample separation. These experiments were successfully conducted after the primary mission objective of separation and collection was successfully completed." (5)

The only other discussion of post-flight results which could be located reported that "...preflight levels of cleanliness were maintained." (2, p. 32)

No further information concerning this experiment could be located at this time.

<Note: Not all of the publications listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Particle Dispersion, Particle Mobility, Electric Field, Surface Charge, Convection, Sedimentation, Electroosmosis, Wall Effect, Medical Applications, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids

Number of Samples: nine
Sample Materials: "The primary sample consisted of concentrated cell culture fluid containing the protein erythropoietin. The eight small volume experimental samples consisted generally of the primary cell culture material or hemoglobin with a protein dye." (5)

Container Materials: Samples entered the Lexan™ separation chamber through thin walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Selected individual samples were collected and stored within a cooled polypropylene reservoir.

Experiment/Material Applications:
Please refer to Lanham, STS-014

References/Applicable Publications:
(1) NASA Space Shuttle Mission 51-D Press Kit, April 1985, p. 14. (preflight)

(2) NASA Space Shuttle Mission 61-B Press Kit, November 1985, pp. 30-32. (post-flight 51-D)

(3) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities, Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing Facility)

(4) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

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(8) Richman, D. W.: System for Hydrodynamic Compensation for Electrophoresis Crescent Distortion. U.S. Patent No. 4,383,905 issued May 1983.

(9) Richman, D. W. and Walker, C. D.: Electrophoresis Apparatus with Flow Control. U.S. Patent No. 4,394,246 issued July 19, 1983.

(10) Personal communication with Co-Investigator C. D. Walker, August 1993.

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Experiment Origin: USA
Mission: STS Launch #23, STS-031 (STS 61-B, Atlantis)
Launch Date/Expt. Date: November 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES), Block 3
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES)

This experiment was the seventh in a series of investigations designed by Lanham et al. and McDonnell Douglas Astronautics Company (MDAC) to study Continuous Flow Electrophoresis (CFE) in the space environment (see Lanham, STS-004, STS-006, STS-007, STS-014, STS-023; Scharp, Morrison and Hymer, STS-008 (all in this chapter)).

<Note: Most of the descriptive information concerning this STS-031 experiment was located in a document which was published prior to the flight (Reference (1)). Co-Investigator C. D. Walker verified that the expected inflight experimental procedures reported in Reference (1) were realized on the actual shuttle mission.>

"The objective of this mission is to separate a sufficient quantity of biological material for animal and clinical testing of a breakthrough pharmaceutical....

"The continuous flow electrophoresis device will operate about 175 hours during the 7 day mission. It is expected that about 66 hours of processing time will be necessary to purify the approximately 1 quart of concentrated protein material on board.

"To ensure that the desired hormone is being separated and collected within the fluid modules, once each day [Payload Specialist Charlie] Walker will run a test on a sample material taken from the collection streams. Using assay material carried on board separately, he will test for hormone presence in the fluid." (1, p. 30) "As during missions STS-14 and STS-23 an enzyme-lined immunoabsorbant antibody-antigen method assay, termed ELISA, was used that was specific to erythropoietin. This assay was developed by MDAC." (4)

"Walker will test daily for the presence of contamination. These tests will be made by withdrawing small samples of fluid from five locations and incubating them in vials previously loaded with free-dried reactants....

"After separation of the biological material is complete, Walker will reconfigure the CFES device to permit additional research on the effects that varying sample concentrations have on the efficiency of the process. Several samples of differing concentrations will be tested to determine the optimum concentration ratio of sample to buffer. " (1, pp. 30-32)

Reportedly, "This research supplemented the work performed during STS-23 on the concentration, conductivity and viscosity interactions of sample and buffer fluid within CFES. Six samples were used and again these experiments were successfully conducted after the primary mission objective of separation and collection was successfully completed." (4)

No other post-flight information describing the space results could be located at this time.

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Particle Dispersion, Particle Mobility, Electric Field, Surface Charge, Convection, Sedimentation, Medical Applications, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Sample Purity, Pharmaceutical Applications

Number of Samples: seven

Sample Materials: "The primary sample consisted of concentrated cell culture fluid containing the protein erythropoietin. The sample fluid is derived from effluent of a genetically engineered cell line developed by Codon (Brisbane, California). The six small volume experimental samples consisted generally of the primary cell culture material or human serum albumin with a protein dye." (4)
Container Materials: Samples entered the Lexan™ separation chamber through two thin walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Selected individual samples were collected and stored within a cooled polypropylene reservoir.

Experiment/Material Applications:
Please refer to Lanham, STS-014.

References/Applicable Publications:
(1) NASA Space Shuttle Mission 61-B Press Kit, November 1985, pp. 30-32. (preflight)

(2) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities, Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

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(9) Personal communication with Co-Investigator C. D. Walker, August 1993.

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Experiment Origin: USA
Mission: STS Launch #8, STS-008 (STS 31-D, Challenger)
Launch Date/Expt. Date: August 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES), Block 2
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES) Pituitary Cell Separation

This experiment was one of three investigations which used the McDonnell Douglas Astronautics Company (MDAC) Continuous Flow Electrophoresis System (CFES) during the STS-008 mission (see also Morrison, STS-008, Scharp, STS-008 (this chapter)). (Other earlier MDAC CFES experiments can be found under Lanham, STS-004, STS-006, STS-007 (also this chapter).) The specific objective of this experiment was to separate subfractions of specific rat pituitary cells (those cells that secrete the growth hormone prolactin and other hormones) from a broader suspension of rat pituitary cells.

Two separate electrophoretic runs were performed in the CFES during this experiment. Prior to a run, a crewman removed syringes containing the prepared cell samples from a 4 ^{o}C cooling storage facility, "...shook it vigorously, and rapidly passed a bar magnet back and forth over the syringe barrel to suspend the cells uniformly." (1, p. 68) After the crewman inserted the syringe into the CFES unit, the sample was automatically ejected from the syringe into the buffer liquid. The sample, which was exposed to an electric field strength of approximately 26 V/cm, resided in the electrophoresis chamber for 12 minutes. After

electrophoretic separation, the fractioned cells were collected from several outlet ports of the CFES, delivered to the collection bags, then placed in the 4 ^{o}C storage facility.

Reportedly, problems associated with the automatic, sample-injection mechanism resulted in a reduction of cells actually separated during each run. In addition, post flight analysis indicated that preflight microbial contamination of the CFES unit resulted in contamination of the collected fractions. However, it was reported that "The pituitary cells were separated into 48 fractions in each of the two electrophoretic runs, and the amounts of growth hormone (GH) and prolactin (PRL) released in to the medium for each cell fraction were determined. Cell fractions were grouped into eight mobility classes and immunocytochemically assayed for the presence of GH, PRL, ...[and other hormones]. The patterns of the hormone distribution indicate that the specialized cells producing GH and PRL are isolatable due to the differences in electrophoretic mobilities." (1, p. 67)

A more detailed discussion of the results of this experiment can be found in Reference (2).

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Particle Dispersion, Particle Mobility, Electric Field, Surface Charge, Convection, Sedimentation, Medical Applications, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Contamination Source, Refrigeration, Hardware Malfunction, Processing Difficulties

Number of Samples: two experiment runs
Sample Materials: live pituitary rat cells
Container Materials: "Samples entered the Lexan^{TM} separation chamber through two thin-walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon^{TM} tubes. Individual fraction tubes, selected preflight, were collected and the samples stored in vinyl receptacles partially prefilled with tissue culture media within the collection cassettes."(8)

Experiment/Material Applications:
"Sample concentrations of proteins and other macromolecules are greatly limited in earth-based separations because of fluid instability which occurs with differences in density between the sample stream and the carrier buffer." (1, p. 67) It was anticipated that in the low-gravity environment (where convective mixing and sedimentation effects are reduced) larger quantities of cell fractions could be produced for subsequent study on Earth.

References/Applicable Publications:
(1) Morrison, D. R., Barlow, G. H., Cleveland, C., Farrington, M. A., Grindeland, R., Hatfield, J. M., Hymer, W. C., Lanham, J. W., Lewis, M. L., Nachtwey, D. S., Todd, P., and Wilfinger, W.: Electrophoretic Separation of Kidney and Pituitary Cells on STS-8. Advances in Space Research, Vol. 4, No. 5, 1984, pp. 67-76. (post-flight)

(2) Hymer, W. C., Barlow, G. H., Blaisdell, S. J., Cleveland, C., Farrington, M. A., Feldmeier, M., Grindeland, R., Hatfield, J. M., Lanham, J. W., Lewis, M. L., Morrison, D. R., Olack, B. J., Richman, D. W., Rose, J., Scharp, D. W., Snyder, R. S., Swanson, C. A., Todd, P., and Wilfinger, W.: Continuous Flow Electrophoretic Separation of Proteins and Cells from Mammalian Tissues. Cell Biophysics, Vol. 10, 1987, pp. 61-85. (post-flight)

(3) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities, Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

(4) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(5) Todd, P., Hymer, W. C., Morrison, Dr. R., Goolsby, C. L., Hatfield, J. M., Kunze, M. E., and Motter, K.: Cell Bioprocessing in Space: Applications of Analytical Cytology. In Physiologist, Supplement, Vol. 31, February 1988, pp. S-52 - S-55. (post-flight)

(6) Hymer, W. C., Grindeland, R., Hayes, C., Lanham, J. W., Cleveland, C., Todd, P., and Morrison, D.: Heterogeneity in the Growth Hormone Pituitary Gland "System: of Rats and Humans" Implications to Microgravity Based Research. In NASA, Washington, Microgravity Science and Applications Flight Programs, January - March 1987, Selected Papers, Vol. 1, pp. 47-88.

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(8) Input received from Investigators J. W. Lanham, D. W. Richman, and C. D. Walker, August 1993.

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(10) Richman, D. W.: System for Hydro-Dynamic Compensation for Electrophoresis Crescent Distortion. U.S. Patent No. 4,309,268 issued January 5, 1982.

(11) Richman, D. W. and Rose, A. L.: Electrophoresis Chamber. U.S. Patent No. 4,310,408 issued January 12, 1982.

(12) Snyder, R. S. and Rhodes, P. H.: Electrophoresis Experiments in Microgravity. In Proceedings of AIAA/IKI Microgravity Sciences Symposium, 1991, pp. 205-208.

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Co-Investigator(s): Barlow, G. (2), Lewis, M. L. (3), Todd, P. (4), Lanham, J. W. (5)
Affiliation(s): (1) National Aeronautics and Space Administration (NASA), Johnson pace Center (JSC), Houston, Texas; (2) During STS-008: Michael Reese Institute, Chicago, Illinois, Currently: Deceased; (3) During STS-008: NASA Johnson Space Center (JSC), Houston, Texas, Currently: University of Alabama in Huntsville; (4) During STS-008: Pennsylvania State University, University Park, Pennsylvania, Currently: University of Colorado Department of Chemical Engineering, Boulder, Colorado; (5) During STS-008: McDonnell Douglas Astronautics Company, St. Louis, Missouri, Currently: McDonnell Douglas Aerospace, Center for Cell Research, Penn State University, State College, Pennsylvania

Experiment Origin: USA
Mission: STS Launch #8, STS-008 (STS 31-D, Challenger)
Launch Date/Expt. Date: August 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES), Block 2
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES): Kidney Cell Separation

This experiment was one of three investigations which used the McDonnell Douglas Astronautics Company (MDAC) Continuous Flow Electrophoresis System (CFES) during the STS-008 mission (see also Hymer, STS-008, Scharp, STS-008 (this chapter)). (Earlier MDAC CFES experiments can be found under Lanham, STS-004, STS-006, and STS-007 (also this chapter).) The specific objective of this experiment was to separate subfractions of specific kidney cells (those which produce the largest amounts of urokinase) from a broader suspension of cultured primary human embryonic kidney cells.

Two separate electrophoretic runs were performed in the CFES during this experiment. Prior to a run, a crewman removed syringes containing the prepared cell samples from a 4 ^{o}C cooling storage facility, "...shook it vigorously, and rapidly passed a bar magnet back and forth over the syringe barrel to suspend the cells uniformly." (1, p. 68) After the crewman inserted the syringe into the CFES unit, the sample was automatically ejected from the syringe into the buffer liquid. The sample, which was exposed to an electric field strength of approximately 26 V/cm,

resided in the electrophoresis chamber for 12 minutes. After electrophoretic separation, the fractioned cells were collected from several outlet ports of the CFES, delivered to collection bags, then stored in the 4 ^{o}C storage facility.

Reportedly problems associated with the automatic, sample-injection mechanism resulted in a reduction of cells actually separated during each run. In addition, post-flight analysis indicated that preflight microbial contamination of the CFES unit resulted in contamination of the collected fractions.

Despite these difficulties, it was reported that "Kidney cells were separated into more than 32 fractions in each of the electrophoretic runs. Electrophoretic mobility distributions in flight experiments were spread more than ground... [based control experiments]. Multiple assay methods confirmed that all cultured kidney cell fractions produced some urokinase, and five to six fractions produced significantly more urokinase than the other fractions. Several fractions also produced tissue plasminogen activator." (1, p. 67)

It was noted that even with the experiment anomalies, the CFES separation of kidney cells on STS 31-D "... was a vast improvement over the [kidney cell] results obtained with the static column electrophoresis device flown in 1975 on ASTP [Apollo-Soyuz Test Project (ASTP)], especially since the CFES experiments were performed on live cells without prior and subsequent freezing." (1, p. 70) (This related ASTP experiment is detailed under Allen, ASTP.)

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Particle Dispersion, Particle Mobility, Electric Field, Surface Charge, Convection, Sedimentation, Medical Applications, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Solid/Liquid Interface, Liquid/Liquid Interface, Contained Fluids, Contamination Source, Refrigeration, Blood Clotting, Hardware Malfunction, Processing Difficulties

Number of Samples: two experiment runs
Sample Materials: Live, cultured human embryonic kidney cells; the buffer in the CFES was Triethanolamine-potassium acetate (ph 7.25, conductivity 296 mOsm/L).

Container Materials: "Samples entered the Lexan™ separation chamber through two thin-walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Individual fraction tubes, selected preflight, were collected and the samples stored in vinyl receptacles partially prefilled with tissue culture media within the collection cassettes."(9)

Experiment/Material Applications:
"Various lots of kidney cells... maintained in special culture medium containing little or no serum produce urokinase and other plasminogen activators (PA). These human kidney cells have been separated by ground based continuous flow electrophoresis (CFE) into as many as 31 fractions which produced PA... of these fractions, five or six produced high levels of PA, and some fractions appeared to produce predominantly high or low molecular forms of urokinase." (1, p. 68) It was anticipated that in the low-gravity environment, convective mixing and sedimentation effects would be reduced and larger quantities of cell fractions would be produced for subsequent study on Earth.

Urokinase is "...a kidney plasminogen activator that is used medically for digesting blood clots." (2, p. 77)

References/Applicable Publications:
(1) Morrison, D. R., Barlow, G. H., Cleveland, C., Farrington, M. A., Grindeland, R., Hatfield, J. M., Hymer, W. C., Lanham, J. W., Lewis, M. L., Nachtwey, D. S., Todd, P., and Wilfinger, W.: Electrophoretic Separation of Kidney and Pituitary Cells on STS-8. Advances in Space Research, Vol. 4, No. 5, 1984, pp. 67-76. (post-flight)

(2) Morrison, D. R., Lewis, M. L., Barlow, G. H., Todd, P., Kunze, M. E., Sarnoff, B. E., and Li, Z.: Properties Of Electrophoretic Fractions Of Kidney Cells Separated on Space Shuttle Flight STS-8. Advances in Space Research, Vol. 4, No. 5, 1984, pp. 77-79. (post-flight)

(3) Hymer, W. C., Barlow, G. H., Blaisdell, S. J., Cleveland, C., Farrington, M. A., Feldmeier, M., Grindeland, R., Hatfield, J. M., Lanham, J. W., Lewis, M. L., Morrison, D. R., Olack, B. J., Richman, D. W., Rose, J., Scharp, D. W., Snyder, R. S., Swanson, C. A., Todd, P., and Wilfinger, W.: Continuous Flow Electrophoretic Separation of Proteins and Cells from Mammalian Tissues. Cell Biophysics, Vol. 10, 1987, pp. 61-85. (post-flight)

(4) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities. Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

(5) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(6) Todd, P., Kunze, M. E., Williams, K., Morrision, D. R., Lewis, M. L., and Barlow, G. H.: Morphology of Human Embryonic Kidney Cells in Culture After Space Flight. The Physiologist, Vol. 28, No. 6, Suppl., 1985, pp. S-183 - S-184. (post-flight)

(7) Todd, P., Hymer, W. C., Morrison, D. R., Goolsby, C. L., Hatfield, J. M., Kunze, M. E., and Motter, K.: Cell Bioprocessing in Space: Applications of Analytical Cytology. In Physiologist, Supplement. (post-flight)

(8) Input received from Principal Investigator D. R. Morrison, June 1991 and August 1993.

(9) Input Received from Investigators J. W. Lanham, D. W. Richman, and C. D. Walker, August 1993.

(10) Richman, D. W.: System for Hydro-Dynamic Compensation for Electrophoresis Crescent Distortion. U.S. Patent No. 4,309,268 issued January 5, 1982.

(11) Richman, D. W. and Rose, A. L.: Electrophoresis Chamber. U.S. Patent No. 4,310,408 issued January 12, 1982.

(12) Snyder, R. S. and Rhodes, P. H.: Electrophoresis Experiments in Microgravity. In Proceedings of AIAA/IKI Microgravity Sciences Symposium, 1991, pp. 205-208.

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Co-Investigator(s): Tschopp, A. (4)
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Experiment Origin: USA
Mission: STS Launch #8, STS-008 (STS 31-D, Challenger)
Launch Date/Expt. Date: August 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Carry-On Incubator
Builder of Processing Facility: Swiss Federal Institute of Technology, Zurich, Switzerland

Experiment:
Attachment of Human Embryonic Kidney (HEK) Cells to Microcarrier Beads in Microgravity

The biotechnology industry is presently interested in the separation of proteins and cells by the method of low-gravity electrophoresis. Cell candidates for research by this method are human embryonic kidney (HEK) cells. These cells can produce urokinase, which is an enzyme used for treating thrombosis. The survival of the cells is dependent on their attachment and spreading on a substrate after electrophoretic separation. (The HEK cells are called anchorage-dependent cells.)

The primary objective of this STS-008 experiment was to determine if anchorage-dependent, HEK cells would attach to microcarrier beads under low-gravity conditions. Reportedly, the investigators were concerned that cell attachment would be lessened under low-gravity conditions since cell/bead contact would be based on random collisions.

Because the electrophoretic separation of HEK cells (a) was being performed on this shuttle flight during a separate experiment (see Morrison, Continuous Flow Electrophoresis System: Kidney Cell Separation) and (b) could, in the long term, be performed on the Space Station, this experiment was designed to determine (1) the efficiency of the reduced gravity cell attachment, (2) the degree of cell survivability, and (3) the extent to which the attached cells could be further processed in the low-gravity environment (during extended duration missions). If the cells did not attach to the beads, only short-term missions would prove appropriate to insure the survival of the cells after reduced gravity electrophoretic separation. Secondary objectives of the

experiment included (a) observing fluids mixing in space and (b) evaluating the performance of a carry-on incubator to be used during a future Spacelab mission.

The following materials were used for the experiment:

Cells and Medium: Frozen suspensions of HEK cells were grown in a medium consisting of (a) one part Medium 199, (b) one part MEM alpha, and (c) Dulbeco's Modified Eagle Medium. The medium was supplemented with (a) 1.2 g/l of bactopeptone, (b) 0.02 g/l of folic acid, (c) 0.72 g/l of i-inositol, (d) 0.1 g/l of nicotinic acid, (e) 16.2 g/l $NaHCO_3$, (f) 10% fetal bovine serum, (g) 20 mM HEPES, (h) 100 units/ml of penicillin, and (i) 100 mg/ml of streptomycin sulfate. (See Reference (1) for manufacturers of these materials.)

Buffer and Enzyme Solutions: Calcium- and magnesium-free, phosphate-buffered saline (CMF-PBS) which consisted of (a) 2.65 mM KCl, (b) 1.46 mM KH_2HPO_4, (c) 136.9 mM NaCl, and (d) 8.0 mM Na_2HPO_4. Also added to the CMF-PBS were (a) 0.05% trypsin and (b) 0.05% EDTA. (See Reference (1) for manufacturers of these materials.)

Glutaraldehyde Fixative: The glutaraldehyde solution used was 50% aqueous, ultra-pure TEM grade. This solution was further diluted with Dulbeco's PBS to a concentration of 2.5%.

Microcarriers: The microcarrier beads were Cytodex 3 and were prepared by swelling and hydrating in CMF-PBS. They were sterilized overnight in 70% ethanol. Prior to use, the beads were washed three times in CMF-PBS and once in the culture medium. They were then suspended in the culture medium with a concentration of 30 mg/ml.

The flight experiment was performed in a carry-on incubator. The incubator was mounted in an instrument panel on the Space Shuttle port flight deck. The apparatus was capable of maintaining a temperature of 37 ^{o}C using either batteries or onboard power. The incubator contained (a) four cell culture chambers sealed with a piston, (b) four syringes loaded with the microcarrier beads, and (c) four syringes loaded with the glutaraldehyde fixative. (See Reference (2) for further details concerning the apparatus.)

Five days prior to stowage on the shuttle, live HEK cells were (a) thawed, (b) suspended in the culture medium, and (c) placed in 75 cm^2 surface growth flasks (Corning 25110). The cells subsequently attached to the flasks and grew. Fourteen hours prior to launch, the cells (approximately 90% confluent) were removed from the flask surfaces with trypsin-EDTA. The free floating

cells were suspended in the culture medium (concentration of 464,000 cells/ml) and then 6 ml of the cell suspension was pipetted into each of the four cell culture chambers. The incubator with cultures (heated to 37 ^{o}C using batteries) was installed onboard the shuttle. Control cells for ground-based studies were prepared using the same procedure.

After launch, the temperature of the incubator was maintained at 37 ^{o}C using a spacecraft power source. The experiment was initiated 7.5 hours after launch by injecting the beads into the cell culture chambers. Glutaraldehyde fixative was then injected into samples 1, 2, 3, and 4 at 5 minutes, 3 hours, 13.5 hours, and 24.5 hours, respectively. The incubator was switched off and the samples remained in the chambers until the end of the mission (6 days later). Six hours after landing, the samples were returned to the investigators.

Post-flight examination of the cells attached to the beads revealed that "...the amount of cells adhering to the beads increases with increasing incubation time in both the flight and ground control... [samples]. This clearly demonstrates that in microgravity attachment and spreading occurred normally." (1, p. 291) In fact, even though it was originally speculated that cell attachment would be lessened under low-gravity conditions, the attachment was slightly more efficient in the flight samples. This increase in attachment efficiency of the flight material was attributed to the increased bead surface area available for attachment. (Suspended beads (low-gravity conditions) provide a greater surface area than sedimented beads (1-g conditions).) Most of the cell attachment occurred during the first 3 hours. Cell growth and replication appeared to be normal after attachment. "Unspecified adhesion of cells by covalent attachment through an activation of the Cytodex carriers by glutaraldehyde can be excluded. In fact, a clear kinetic attachment is observed within 24.5 h.... If the attachment would have been unspecific there would not be a difference between the samples at different times." (1, p. 292)

Scanning electron microscopy (SEM) (see Reference (1) for sample preparation details) revealed no significant morphological differences between the low-gravity and 1-g samples. "The fact that the cells are showing in the SEM pictures a clear spreading at 25.5... [hours]... led us to the assumption that the cells were functionally intact. More than 50% of the cells [estimated value from population data] were bound at this time to beads.... The fate of the rest of the cells is unknown since it was not possible for technical reasons to count the free cells and the cells adhered to the walls of the cell culture chambers. Moreover the population of the cells used in this experiment is a rather inhomogeneous one and therefore we cannot assume that they adhere

all in the same manner and at the same time." (1, p. 292)

It was concluded that microcarriers could be used "...in the receptacles containing subpopulations of cells separated by continuous flow electrophoresis. Since the cells are able to attach to microcarriers much more cells would remain viable until they are returned to earth. This technique should avoid the 65-80% decrease in viability observed after electrophoretic separation on STS-8 [see for example Morrison et al., Continuous Flow Electrophoresis System: Kidney Cell Separation] and other flights." (1, p. 292)

Key Words: Biotechnology, Biological Cells, Suspension of Particles, Cell Attachment, Cell Storage, Sedimentation, Liquid Injection, Liquid Expulsion through a Small Orifice, Liquid Mixing, Aqueous Solutions, Solid/Liquid Interface, Contained Fluids, Incubator, Piston System, Electrophoresis, Medical Applications

Number of Samples: four
Sample Materials: See Experiment section
Container Materials: unknown

Experiment/Material Applications:
HEK cells were used for this experiment since they (1) produce the important enzyme urokinase, (2) were simultaneously investigated during the mission for electrophoretic separation (see Morrison et al., Continuous Flow Electrophoresis System: Kidney Cell Separation), and (3) are good models for all types of anchorage-dependent cells.

References/Applicable Publications:
(1) Tschopp, A., Cogoli, A., Lewis, M. L., and Morrison, D. R.: Bioprocessing in Space: Human Cells Attach to Beads in Microgravity. Journal of Biotechnology, Vol. 1, 1984, pp. 287-293. (post-flight)

(2) Cogoli, A. and Tschopp, A.: Biotechnology in Space Laboratories. In Advances in Biochemical Engineering, Vol. 22, pp. 1-50, Springer-Verlag, Berlin, 1982. (preflight, experiment apparatus description)

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Experiment Origin: USA
Mission: STS Launch #8, STS-008 (STS 31-D, Challenger)
Launch Date/Expt. Date: August 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Continuous Flow Electrophoresis System (CFES), Block 2
Builder of Processing Facility: McDonnell Douglas Astronautics Company, St. Louis, Missouri

Experiment:
Continuous Flow Electrophoresis System (CFES) Pancreas Cell Separation

This experiment was the fourth in a series of investigations initiated by McDonnell Douglas Astronautics Company (MDAC) to study Continuous Flow Electrophoresis (CFE) in the space environment (see Lanham, STS-004, STS-006, STS-007 (this chapter)). This experiment was one of three experiments which used the Continuous Flow Electrophoresis System (CFES) during the STS-008 mission (see Morrison, STS-008; Hymer, STS-008 (also this chapter)).

These MDAC/Washington University School of Medicine experiments examined the electrophoretic separation of canine pancreatic cells. During the investigation, two electrophoretic runs were realized using the CFES. Reportedly, the specific experiment procedures during these runs were similar to the other two experiments performed using the CFES during the mission (see Morrison, STS-008 or Hymer, STS-008).

After the mission it was reported that "...preflight microbial contamination of the CFES was not completely inhibited by preflight chemical sterilization... <and these experiments> had some degree of contamination." (2, p. 70) No documentation detailing post-flight results has been located.

<Note: Not all of the references listed below were available at the time this experiment summary was prepared.>

Key Words: Biotechnology, Continuous Flow Electrophoresis, Electrophoresis, Separation of Components, Biological Cells, Particle Dispersion, Particle Mobility, Electric Field, Surface Charge, Convection, Sedimentation, Medical Applications, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Contained Fluids, Contamination Source

Number of Samples: two electrophoretic runs
Sample Materials: live canine pancreatic cells
Container Materials: "Samples entered the Lexan™ separation chamber through two thin-walled glass tubes. All fractioned samples and carrier buffer were isolated and exited the separation chamber through an array of 197 Tygon™ tubes. Individual fraction tubes, selected preflight, were collected and the samples stored in vinyl receptacles partially prefilled with tissue culture media within the collection cassettes."(5)

Experiment/Material Applications:
"Four major types of endocrine cells are found in the pancreatic islets. Beta cells produce insulin, alpha cells produce glucagon, delta cells produce somatostatin, and F cells produce pancreatic polypeptide.... Purified populations of the four types of islet cells, if they could be made available would greatly benefit the research on the role of specific cell-cell interactions in normal and abnormal islet physiology..., the role of the islet antibodies in diabetes..., and the development of pure islet cell populations for transplantation into diabetic patients." (1, pp. 78-79)

References/Applicable Publications:
(1) Hymer, W. C., Barlow, G. H., Blaisdell, S. J., Cleveland, C., Farrington, M. A., Feldmeier, M., Grindeland, R., Hatfield, J. M., Lanham, J. W., Lewis, M. L., Morrison, D. R., Olack, B. J., Richman, E. W., Rose, J., Scharp, D. W., Snyder, R. S., Swanson, C. A., Todd, P., and Wilfinger, W.: Continuous Flow Electrophoretic Separation of Proteins and Cells from Mammalian Tissues. Cell Biophysics, Vol. 10, 1987, pp. 61-85. (postflight)

(2) Morrison, D. R., Barlow, G. H., Cleveland, C., Farrington, M. A., Grindeland, R., Hatfield, J. M., Hymer, W. C., Lanham, J. W., Lewis, M. L., Nachtwey, D. S., Todd, P., and Wilfinger, W.: Electrophoretic Separation of Kidney and Pituitary Cells on STS-8. Adv. Space Research, Vol. 4, No. 5, pp. 67-76, 1984. (post-flight; mentions pancreatic cell research)

(3) Continuous Flow Electrophoresis System. In Microgravity Science and Applications Experiment Apparatus and Facilities. Document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 12. (processing facility)

(4) Naumann, R. J.: Microgravity Science and Applications. In In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 18-19. (post-flight; very short description)

(5) Input Received from Investigators J. W. Lanham, D. W. Richman, and C. D. Walker, August 1993.

(6) Richman, D. W.: System for Hydro-Dynamic Compensation for Electrophoresis Crescent Distortion. U.S. Patent No. 4,309,268 issued January 5, 1982.

(7) Richman, D. W. and Rose, A. L.: Electrophoresis Chamber. U.S. Patent No. 4,310,408 issued January 12, 1982.

(8) Snyder, R. S. and Rhodes, P. H.: Electrophoresis Experiments in Microgravity. In Proceedings of AIAA/IKI Microgravity Sciences Symposium, 1991, pp. 205-208.

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<Note: The current affiliation of Cogoli is unclear. In 1989 he indicated that he was still at the Federal Institute of Technology. However, it appears he may now be at the Space Biology Group, ETH Technopark, Zurich, Switzerland.>

Experiment Origin: Switzerland
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack #4
Processing Facility: cell culture incubators, cell culture containers
Builder of Processing Facility: Federal Institute of Technology, Zurich, Switzerland

Experiment:
Lymphocyte Proliferation in Weightlessness (1ES031)

"Lymphocytes constitute about 30% of the white cells in human blood and are important in maintaining immunity against infection." (12, p. 9) Upon specific recognition of a foreign substance (called an antigen), lymphocytes undergo transformation from a "resting" into an active status: cells begin to grow and proliferate and secrete antibodies and lymphokines. This transformation can be simulated in cultures of purified lymphocytes which have been exposed to mitogens (cell activators) like Concanavalin A (Con A). The extent of the activation can be measured by the amount of tritiated thymidine incorporated into DNA.

This Spacelab experiment was the first in a series of investigations designed by Cogoli et al. to determine the effect of a reduced gravity environment on the activation and differentiation of human lymphocytes in-vitro.

In preparation for the experiment, cultures of lymphocytes were prepared 5 hours prior to shuttle launch. During the mission, the cultures were held in a 37 ^{o}C incubator and exposed to Con A for 72$_3$ hours. The exposure was followed by a 2-hour treatment with ^{3}H-Thymidine. The samples were then treated with Hydroxyethyl Starch (HES) as a cryopreservative and frozen in liquid nitrogen.

Post-flight analysis of the response of the lymphocytes revealed that activation was depressed by more than 90% compared to synchronous ground samples. Speculation as to why such a marked depression resulted was not detailed.

<Note: Most of the publications listed below were not available to aid in the preparation of this experiment summary. It is suspected that the reason for the activation depression is detailed in one or several of these documents.>

Key Words: Biotechnology, Biological Cells, White Cells, Lymphokines, Lymphocyte Activation, Cell Differentiation, Particle Dispersion, Particle Motion, Cell Storage, Sample Purity, Medical Applications, Immune System, Antigen, Mitogen, Antibodies, Incubator, Freezing, Pharmaceutical Applications

Number of Samples: four flasks
Sample Materials: White blood cells (human lymphocytes), human serum; Cell culture medium: RPMI1640; Additives: (1) mitogen: concanavalin A, (2) ^{3}H-Thymidine, and (3) HES-hydroxyethyl starch
Container Materials: Cell culture flashes: Teflon^{TM}/glass fiber

Experiment/Material Applications:
Lymphocyte activation involves intercellular cell contacts, i.e., movements of particles (outer diameter = 7-15 micrometers) in a fluid, as well as intracellular streaming of cytoplasm and organelles. Secretion products like alpha and gamma-interferron as well as other lymphokines are of great pharmaceutical relevance. As indicated by the experimental results, the low-gravity environment reduces the cellular reactivity. Similar "Changes in the body's immune response would be important on a long mission in space." (12, p. 9)

References/Applicable Publications:
(1) Cogoli, A., Tschopp, A., and Fuchs-Bislin, P.: Cell Sensitivity to Gravity. Science, 225, 1984, pp. 228-230. (post-flight)

(2) Tschopp, A. and Cogoli, A.: Low Gravity Lowers Immunity to Diseases. New Scientist, August 23, 1984, p. 36. (post-flight)

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(4) Cogoli, A., Tschopp, A., and Fuchs-Bislin, P.: Experiment 1ES031 on Spacelab-1: Are Cells Sensitive to Gravity? In Life Sciences Research in Space, ESA SP-212, 1984, pp. 19-22. (post-flight)

(5) Cogoli, A. and Tschopp, A.: Lymphocyte Reactivity During Spaceflight. Immunology Today, Vol. 6, pp. 1-4, 1985. (post-flight)

(6) Cogoli, A.: Sensitivity of Human Lymphocytes to Microgravity in vitro. In AGARD Conference Proceedings No. 377, AGARD-NATO, Neuilly-sur-Seine, France, 1985, pp. 11-1 - 11-8. (post-flight)

(7) Cogoli, A.: Weltraumbiologie: Das berhalten der Zellen in Mikrogravität. In Vierteljahrsschrift der Naturforschenden Gesellschaft in Zürich, Vol. 130, 1985, pp. 334-336. (post-flight)

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(10) Gmünder, F. K. and Cogoli, A.: Cultivation of Single Cells in Space. Appl. Micrograv. Tech., Vol. 1, 1988, pp. 115-122. (post-flight; includes information related to ground based work)

(11) Cogoli, A.: Space Biologist's Inflight Safety Considerations. Space Safety and Rescue 1986-1987, Vol. 70, 1988, pp. 217-221. (post-flight, includes information related to ground based work and D1)

(12) Effect of Weightlessness on Lymphocyte Proliferation 1ES031 (ESA). In Spacelab 1 Experiments, NASA Marshall Space Flight Center, 15M983, p. 9.(preflight)

(13) Input received from Principal Investigator A. Cogoli, October 1989.

<Note: For additional publications related to post-flight Spacelab 1 results, see Cogoli, Spacelab D1, publications listing.

Many other documents related to this experiment have been written. Most describe preflight and ground-based efforts. Due to space limitations they were not listed here.>

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Co-Investigator(s): Bechler, B. (2), Müller, O. (3)
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<Note: The current affiliation of Cogoli is unclear. In 1989 he indicated that he was still at the Federal Institute of Technology. However, it appears he may now be at the Space Biology Group, ETH Technopark, Zurich Switzerland.>

Experiment Origin: Switzerland
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility: Biorack; Rack #5
Processing Facility: Special blood kit, cell culture containers, syringe containers, two cell culture incubators, and reference centrifuges
Builder of Processing Facility: Blood kit: Cogoli, M., Zurich, Switzerland; containers: Swiss Federal Institute of Technology, Zurich, Switzerland

Experiment:
Lymphocyte Activation (BR 32/33 CH)

This Spacelab D1 experiment was the second in a series of investigations designed by Cogoli et al. to determine the effect of a reduced gravity environment on the activation and differentiation of human lymphocytes (see Cogoli, STS-009). The D1 investigation consisted of two experiments designated as (1) "Blood" and (2) "Lympho."

Blood Experiment

The specific objective of the first experiment ("Blood") was to study the effect of space flight stress and space flight conditions on crewman lymphocyte activation. Whole blood samples were drawn from four astronauts (ex-vivo) before, during, and after the flight. Each participating crewman (designated as A, B, C, or D) had the following blood types: Crewman A (A, Rhesus +); Crewman B (O+); Crewman C (O+); and Crewman D (AB-). "10 ml of blood was drawn from each subject at the following times: 9 days... and 2 days... prior to flight, 1 hour after landing... and 7 and 13 days... after landing. In-flight withdrawals (5 ml from each subject) were made 48 and 60 hours after launch...." (1, p. 367). <Note: Details of blood handling after collection is

not presented here but is described in Reference (1), p. 367.>

While the earlier Spacelab 1 lymphocyte activation experiment (See Cogoli, STS-009) was not equipped with onboard reference centrifuges (for in-flight comparison of 1Xg centrifuged shuttle samples to low-g (Oxg) shuttle samples) this D1 experiment was equipped with reference centrifuges. The experimental setup also consisted of (1) a blood kit (to enable the drawing and storing of blood), (2) two incubators (one at 37 ^{o}C and one at 22 ^{o}C), and (3) cell culture containers. "On the ground, 3 culture flasks were prepared for each donor and for each withdrawal, one without and two with [mitogen concanavalin a, (con A)]. In flight, two cultures per donor were prepared, both with con A, one maintained under microg conditions, the other at 1Xg in the centrifuge." (1, p. 367) Cultures which were exposed to con A, were exposed for 72 hours, then treated with ^{3}H-thymidine. Other treatments were described in Reference (1), p. 367. The extent of the activation was measured by the amount of tritiated thymidine incorporated into DNA.

Reportedly, the activation of the lymphocytes from the four crew members was significantly depressed immediately following the flight. Lymphocyte activation returned to normal 7 days after landing. Thus, it was surmised that the reduced lymphocyte activation was due to physical and psychological stress rather than to microgravity conditions.

"From a cellular point of view the most important implication of our findings is that lymphocytes maintained at O-g in flight are not activated in contrast to the in flight 1-g controls. This coincides with the surprising data of the experiment performed on Spacelab-1... and also with the data of the Lympho experiment described below." (1, p. 370)

Lympho Experiment

The specific objective of the second experiment ("Lympho") was similar to the objective of the Spacelab 1 experiment: to determine if the microgravity environment depressed lymphocyte activation in-vitro. Shortly before flight, cultures of human lymphocytes were purified. "16 culture flasks were each filled with 8 ml of culture medium containing the cells. One set of 8... [flasks] was retained for the ground control experiment in a biorack identical to the flight unit. The ground centrifuge generated a 1.4Xg environment. The other 8 flasks were delivered for installation on board Spacelab 12 hours before launch. 12 hours after launch, con A (25... [micrograms]/ml) was injected into all cultures. After injecting the mitogen, four cultures of each set (flight and ground) were transferred to the incubator

and four were fastened to the rotor of the centrifuge. After 36, 48, 72 and 96 h of incubation with con A, 4 cultures (2 in flight and 2 on the ground, one from the incubator and one from the centrifuge) were treated with tritiated thymidine (2 μCi/ml)...." (1, p. 368)

Analysis of the "Lympho" experiment confirmed the results of Spacelab 1. Again, activation (of the non-centrifuged samples) was depressed by more than 90%.

It was noted that Earth samples had higher activity than space flight 1Xg centrifuge samples. Several possible reasons for the higher activity were cited:

(1) Damage due to cosmic radiation may have taken place in the space cells thus reducing the activation.

(2) "The reference centrifuge installed in the 37 ^{o}C incubator hosted a number of other investigations each requiring one or more stops during operations. At the end of the mission the total 'stopping time' was found to be 50 minutes. Each stop is equivalent to a microgravity exposure for the cells." (1, p. 371)

(3) A 1 ^{o}C temperature difference between flight and ground incubators may have affected the results.

Analysis of the samples by electron microscopy indicated remarkable differences between low-gravity and one-gravity samples. "In microgravity conditions, lymphocytes did not form the typical aggregates as seen in the presence of con A at 1Xg. However, clusters of cells resembling monocytes were seen in microg- which show numerous vacuoles containing engulfed material resulting from extensive cell death." (1, p. 366 (abstract))

<Note: Several of the publications listed below were not available to aid in the preparation of this experiment summary.>

Key Words: Biotechnology, Biological Cells, White Cells, Lymphokines, Lymphocyte Activation, Cell Differentiation, Liquid Injection, Liquid Expulsion Through a Small Orifice, Centrifuge, Incubator, Cell Storage, Cell Survivability, Particle Dispersion, Particle Aggregation, Medical Applications, Immune System, Antigen, Mitogen, Antibodies, Cosmic Rays

Number of Samples: Several; see above experiment summary.
Sample Materials: White blood cells (human lymphocytes), human serum; Culture Medium: RPMI1640; Additives: con A (mitogen), ^{3}H-thymidine, glutaraldehyde
Container Materials: Teflon^{TM}/glass
Experiment/Material Applications:
Please see Cogoli, Spacelab 1.

References/Applicable Publications:

(1) Cogoli, A., Bechler, B., Müller, O., and Hunzinger, E.: Effect of Microgravity on Lymphocyte Activation. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 366-375. (post-flight)

(2) Bechler, B. and Cogoli, A.: Lymphozyten sind schwerkrafttempfindlich. In Natur wissenschaften, Vol. 73, 1986, pp. 400-403. (post-flight; in German)

(3) Cogoli, A.: Effect of Spaceflight on Lymphocyte Proliferation. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 155-157. (preflight)

(4) Cogoli, A. and Müller, O.: Lymphocytes are Sensitive to Gravity. In Scientific Results of the German Spacelab Mission D1 (abstracts), Norderney, Germany, August 27-29, 1986, pp. 6-7. (post-flight)

(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements on D1)

(6) Cogoli, A.: Plädoyer für die bemannte Raumfahrt, Bild der Wissenschaft, Vol. 5, 1986, pp. 136-143. (post-flight; in German)

(7) Lorenzi, G., Fuchs-Bislin, P., and Cogoli, A.: Effects of Hypergravity on "Whole-blood" Cultures of Human Lymphocytes. Aviat. Space Environ. Med., Vol. 57, 1986, pp. 1131-1135. (post-flight and ground-based research)

(8) Cogoli, A., Bechler, B., Lorenzi, G., Gmünder, F., and Cogoli, M.: Cell Cultures in Space: From Basic Research to Biotechnology. In Biological Sciences in Space, eds., Watanabe, S., Mitaray, G., and Mori, S., Myu Research, Tokyo, 1987, pp. 225-232. (post-flight)

(9) Cogoli, A.: Cell Cultures in Space: From Basic Research to Biotechnology II. In Life Sciences Research in Space, ESA SP-271, 1988, pp. 285-290. (post-flight; includes results from Spacelab 1 and STS-008)

(10) Cogoli, A., Bechler, B., Müller, O., and Hunzinger, E.: Effect of Microgravity on Lymphocyte Activation, In Biorack on Spacelab D1. eds. Longdon, N. and David, V., ESA SP-1091, 1988, pp. 89-100. (post-flight)

(11) Cogoli, A., Gmünder, F. K., and Nordau, C. G.: Cell Biology in Space: From Basic Science to Biotechnology III. In Space Commerce, Proceedings of the 2nd International Conference and Exhibition on the Commercial and Industrial Uses of Outer Space. Gordon and Breach Science Publishers, New York-London-Paris-Montreux-Tokyo-Melbourne, 1988, pp. 353-366. (post-flight)

(12) Input received from Principal Investigator A. Cogoli, October 1989.

<Note: For additional publications related to post-flight Spacelab D1 results, see Cogoli, Spacelab 1 publication listing.

Many other documents which are related to this experiment are available and describe preflight and ground-based efforts. Due to space limitations they were not printed here.>

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Experiment Origin: Unknown, either (1) USA and Canada or (2) USA or Canada
Mission: STS Launch #16, STS-023 (STS 51-D, Discovery)
Launch Date/Expt. Date: April 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment: handheld experiment, mounted onto standard light box on middeck
Processing Facility: Phase Partitioning Experiment (PPE) Unit: fifteen-chambered experimental module
Builder of Processing Facility: Campbell Engineering, Huntsville, Alabama

Experiment:
Phase Partitioning Experiment (PPE)

"Phase partitioning is a selective, yet gentle and inexpensive technique, ideal for the separation of biomedical materials such as proteins. It involves establishing a two-phase system by adding various polymers to a water solution containing the materials to be separated.... When two phase polymer systems are established, the biomedical materials they contain tend to separate or 'partition' into different phases.

"Theoretically, phase partitioning should separate cells with significantly higher resolution than is presently obtained in the laboratory. It is believed that when the phases are emulsified on Earth, the rapid gravity driven fluid movements occurring as the phases coalesce tend to randomize the separation process. It is expected that the theoretical capabilities of phase partitioning systems can be more closely approached in the weightlessness of orbital spacecraft where gravitational effects of buoyancy and sedimentation are minimized." (8, p. 17)

This STS 51-D experiment was the first in a series of investigations designed by Brooks et al. to study the low-gravity demixing of aqueous, polymer two-phase systems. The overall objective of the experiment was to evaluate phase partitioning when the driving forces of convection and sedimentation were absent.

An earlier immiscible liquids experiment performed during Skylab (see Lacy, Skylab SL-4) demonstrated that the employed Skylab oil and water system (which formed an unstable emulsion on Earth) formed a stable emulsion in space, and during several hours, illustrated no demixing. Therefore, a major objective of this low-gravity phase partitioning experiment was to determine if the two polymer phase systems would indeed "...demix and become localized in an acceptable length of time." (4, p. 9)

The experiment was performed in a small plastic box which contained 15 transparent chambers. Each chamber (1.5 X 1.8 X 1.0 cm) contained a stainless steel mixing ball and either (a) a dextran/PEG system or (b) an isopycnic system of dextran/PEG/Ficoll (this mixture was used as a reference material). It appears that six systems were examined during the mission. (See the sample materials section below for details, or for more information, Reference (5), p. 132.)

Reportedly, "At the beginning of the experiment, the box was shaken manually, the action of the steel balls being sufficient to emulsify each sample. The box was then taped to a fluorescent light box and photographed at appropriate times over a period of 110 minutes to record the evolution of the demixing." (5, p. 134)

After the shuttle mission, photos taken during the space experiment were compared with photos taken during similarly performed ground-based control experiments. The ground control experiments (which employed isopycnic mixtures of dextran/PEG/Ficoll) demixed within approximately 10 minutes following mechanical mixing (at constant temperature). "Microscopically, domains of each phase could be detected 2 s after mixing. As the domains grew, reducing the area of interface, they formed three dimensional bicontinuous structures which eventually evolved into a central, dextran-rich phase surrounded by the Ficoll-PEG phase." (5, p. 133)

In the space experiments, "It... [was] clear that after only 19 s some degree of demixing was present. Ten minutes after agitation large areas of each phase... [had]... become localized in three of the systems although... [the 6% dextran/4% PEG system] showed no appreciable demixing, presumably because of the combination of high viscosities and interfacial tension in this more concentrated system.

"The other interesting feature seen... [was]... the tendency for the PEG-rich phase, dyed dark by including Trypan Blue in each sample, to occupy the regions near the chamber walls, the dextran-rich (light) phase forming spheres in the interiors of the containers. This tendency became even more pronounced at longer times.... This distribution occurs because the PEG rich

phase has a higher affinity for the chamber wall than does the other phase. Hence it tends to preferentially wet the boundaries and displace the dextran rich phase." (4, p. 10) "The steel mixing balls, on which the dextran rich phase spread, were found within that phase in every case." (5, p. 136)

The photos indicated (qualitatively), "...in all cases demixing occurred more rapidly on the ground than in space. However the systems... [5% Dextran/3.5% PEG, 5% Dextran/4.0% PEG, and 7% Dextran/0.29% PEG/12% Ficoll]... characterized by values of interfacial tension and viscosities typical of those useful for cell partitioning on earth demixed reasonably rapidly. Systems comprised of higher polymer concentrations, with larger differences in concentration between the phases... [6% Dextran/4% PEG, 8% Dextran/4% PEG], were much slower to demix. This phase volume also had a strong effect on the demixing rate in the ... [8% Dextran/4% PEG]... system. Interestingly, the chamber with the 1/1 mixture [the isopycnic mixture] did not demix as soon as did that containing the lower amount of PEG-rich phase. In the sample in which this ratio was reversed, no visible mixing occurred over the time course of the experiment." (5, p. 134)

"The presence of particulates, (fixed erythrocytes) had no effect on the demixing rate in one case... [5% Dextran/4% PEG], but appeared to reduce the rate in the other system [7% Dextran/5% PEG]... the reason for the observed behaviour is not clear at present.

"The final noteworthy feature of the PPE-1 experiment is the apparent shift in chemical equilibrium which became evident about 15 to 20 minutes after shaking. Under laboratory conditions, once the phases demix they remain stable and of constant composition indefinitely. The PPE-1 photographs clearly show a change in composition of the localized PEG-rich phases for 30 min onwards, seen as a further decomposition of the fairly uniform dark regions into mottled areas of demixing. We have never observed phenomenon like this on the ground unless a change in the experimental conditions, such as dilution, concentration through evaporation or a temperature shift occurred. In any of these cases, the shift in equilibrium results in a change in composition of each of the phases, presumably in the present instance by the mechanism of spinoidal decomposition since the systems flow lie well within the binodal. It seems very probable that the secondary decomposition observed in the PPE-1 experiment was due to a temperature increase. It was found after the flight that the surface of the light box increased in temperature by 9 ^{o}C in 30 min, the likely source of the temperature change." (5, p. 136)

It was concluded that "The results of this first space experiment were very encouraging then, as it seems clear that demixing of the phases can occur in times which are compatible with cell separation experiments. The rate at which the demixing occurs evidently depends on the system composition, so it can be controlled experimentally. Localization of the demixed phases apparently can also be controlled if the wetting of the chamber walls can be manipulated." (4, pp. 10-11)

A detailed discussion of the possible mechanisms controlling demixing of the system were presented in Reference (5). Also presented in several of the references were the results of related KC-135 experiments.

Key Words: Biotechnology, Phase Partitioning, Phase Separation, Separation of Components, Immiscible Fluids, Aqueous Solutions, Polymers, Two-Phase System, Emulsion, Dispersion, Particle Dispersion, Particle Distribution, Particle Coalescence, Spinodal Decomposition, Liquid/Liquid Dispersion, Droplet Dispersion, Liquid/Liquid Interface, Solid/Liquid Interface, Liquid Mixing, Liquid Demixing, Liquid Stability, Buoyancy Effects Diminished, Buoyancy-Driven Convection, Sedimentation, Density Difference, Contact Angle, Interfacial Tension, Wetting, Wetting of Container, Viscosity, Biological Cells, Thermal Environment More Extreme than Predicted, Contained Fluids

Number of Samples: six
Sample Materials: Dextran/polyethylene glycol(PEG) systems were examined during the mission: (1) 5% dextran 500 (500 indicates molecular weight was 500,000), 3.5 % PEG 8000, 0.1M phosphate buffer, pH 7.2, (2) 5% dextran 500, 4% PEG 8000, 0.1M phosphate, (3) 6% dextran 500, 4% PEG 8000, 0.1M phosphate, (4) 8% dextran, 4% PEG, (5) 7% dextran 40, 5% PEG and (6) 7% dextran 500, 0.29 PEG 8000, 12% Ficoll, 0.1M phosphate.
Container Materials: Plastic box with stainless steel mixing ball. <Note: It is not clear if the fluids were in contact with the plastic or some other material.>

Experiment/Material Applications:
"The... [principal] biotechnological interest in aqueous phase partitioning at present is its application in the isolation of macromolecules. However, the preparation of pure subpopulations

of viable cells from the complex mixtures in which they are found is also a problem in a number of areas of commercial interest, the isolation of insulin-producing cells for the development of an... [unreadable] artificial pancreas being one example.... Such applications require high resolution gentle procedures, characteristics which are a feature of the partitioning process." (4, p. 2)

References/Applicable Publications:

(1) Van Alstine, J. M., Karr, L. J., Harris, J. M., Snyder, R. S., Bamberger, S. B., Matsos, H. C., Curreri, P. A., Boyce, J., and Brooks, D. E.: Phase Partitioning in Space and on Earth. In Immunobiology of Proteins and Peptides, IV: T-Cell Recognition and Antigen Presentation, M. V. Atassi, Ed., Plenum Press, New York, pp. 307-328 (1988).

(2) Brooks, D. E., Bamberger, S. B., Harris, J. M., and Van Alstine, J.: Rationale for Two Phase Polymer Systems in Microgravity Separation Experiments. In Proceedings 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 315-318 (1984). (preflight)

(3) Van Alstine, J., Harris, J. M, Snyder, S., Curreri, P. A., Bamberger, S., and Brooks, D. E.: Separation of Aqueous Two-Phase Polymer Systems in Microgravity. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 309-313. (Related KC-135 work)

(4) Brooks, D. E., Boyce, J., Bamberger, S. B., Harris, J. M., and Van Alstine, J. M.: Separation of Biological Materials in Microgravity. NASA TM-101142, 15 pp. (post-flight)

(5) Brooks, D. E., Bamberger, S. B., and Harris, J. M.: Demixing Kinetics of Phase Separated Polymer Solutions in Microgravity. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 131-138 (1987).

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(7) Karr, L. J.: Phase Partitioning. Research and Technology 1987, Annual Report of the Marshall Space Flight Center, pp. 33-34. (ground-based-related research)

(8) NASA STS 51-D Press Kit, pp. 17-18. (preflight)

(9) Van Alstine, J. M., Bamberger, S., Harris, J. M., Snyder, R. S., Boyce, J. F., and Brooks, D. E.: Phase Partitioning Experiments on Shuttle Flight STS-26. In Seventh European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-295 (January 1990), pp. 399-407. (post-flight; includes the STS 51-D results)

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Experiment Origin: Unknown, (USA and/or Canada)
Mission: STS Launch #26, STS-26 (Discovery)
Launch Date/Expt. Date: October 1988
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment: handheld experiment, stowed in middeck locker
Processing Facility: Module with eighten chambers filled with small quantities of two-phase systems
Builder of Processing Facility: Unknown

Experiment:
Phase Partitioning Experiment (PPE)

This STS-26 experiment was the second in a series of investigations designed by Brooks et al. to study the low-gravity demixing of aqueous, polymer two-phase systems (see Brooks, STS-023). A discussion of the phase partitioning technique can be found under Brooks, STS-023.

The STS-26 experiment apparatus "...consisted of a... [Plexiglas™] body plate into which 18 chambers were machined.... Offset fill ports and O-rings allowed for clear photo visualization between the [partitioned] phases and the chamber wall. Each chamber contained a mixing ball.... A... [+ or -] 1 ^{o}C liquid crystal temperature strip was imbedded in the unit as part of the photo field. Chambers 1 to 4, 6, and 9 to 12 were the same shape as on... [STS-023]. They had dimensions of 1.90 cm by 1.57 cm wide by 0.91 cm deep and held 3.4 ml. Chamber 5 was 1.40 cm deep and held 4.2 ml. Chambers 7, 8, and 13-18 were 1.40 cm cubes to allow the non-wetting demixed phase to form a sphere.... The latter six chambers held quartz cuvettes which were fully or half-coated with dextran... or polyacrylamide...." (4, p. 401) Sixteen of the chambers contained mixtures of dextran (D), polyethylene glycol (PEG), and ficoll. The remaining two chambers contained mixtures of FC43 fluorocarbon oil and water. (A complete listing of the material systems flown during this experiment can be located in Reference (4).)

The phase partitioning experiment was performed a total of four times during the STS-026 Mission. During each of these four runs, "The unit was mixed by manual agitation and then mounted with a light diffuser on an orbiter middeck fluorescent light box via velcro standoffs also acted to thermally isolate the unit." (4, p. 401) Video recording, 35 mm photography, and voice recordings provided data.

Reportedly, "Although the experiment was designed to be performed at 22 ^{o}C it was actually performed at 27 ^{o}C to 31 ^{o}C due to problems with the shuttle cooling system. However, during each experiment the temperature varied at most... [+ or -] 2^{o}C." (4, p. 403)

Post-flight visual inspection of the photographs indicated that material systems demixed at rates which varied with the polymer composition and chamber dimensions. This behavior was somewhat analogous to similar 1-g experiments. "In both environments when the more viscous phase wet the container wall the demixing took place more slowly. The viscosity of the non-wetting, dispersed phase appeared to play very little role in the process, at least when it was the more viscous of the two." (4, p. 405) This was illustrated in chambers 7 and 8 where the system which demixed more rapidly had the higher dispersed phase viscosity and lower suspending phase viscosity.

Chamber size became critical later in the demixing process, when domain size became significant with respect to the chamber dimensions. "Chambers 4, 5 and 8 contained the same system but had different aspect ratios. In one g, chamber 5 (1.9 cm height x 1.57 cm width x 1.4 cm depth) demixed more slowly than 4 (1.9 cm x 1.57 cm x 0.91 cm) or 8 (1.4 cm^3). In low g the cubical chamber demixed the most rapidly in the latter stages. This occurred because locally the domains grew at the same nominal rate in all three chambers. As the thickness of the wetting layer contacting the wall increased[,] the interior D-rich phase collected and individual regions increased in size, accumulating into a single central 'yoke'. In the cubical chamber this accumulation occurred earlier than in the non-cubical geometries because the linear dimensions through which the interior regions had to grow before interacting were minimized in this shape." (4, p. 405)

The effects of volume fraction on demixing were also investigated. It was reported that, on Earth, the material "...in which the lower viscosity, wall wetting phase dominated in volume fraction[,] demixed faster than that in which the more viscous interior phase was at the higher volume ratio." <Note: It is not clear from the available references what the lower & higher viscous materials were.> (4, p. 405) This result was also observed under low-gravity conditions. In space, this type of system

demixed faster than one with a 1:1 ratio. On Earth, the 1:1 ratio sample separated faster than any other system. "Again it would seem that the thickness and viscosity of the phase in contact with the walls can be rate limiting. Whatever the mechanism by which the domains of interior phase accumulate into a single central volume, viscous dissipation in the suspending medium evidently plays an important role." (4, p. 405)

Plots of a characteristic dimension vs. time were constructed for quantitative analysis (see Reference (4) for analysis details). A log-log plot of this data revealed a linear dependence of average domain size with demixing time. Results from a single (earlier) STS-023 experiment "...supported a coalescence mechanism, based largely on the increased rate seen when the interior phase was at a higher volume concentration than the [wall] wetting phase. This was interpreted as producing an increased collision frequency and coalescence rate between interior drops. That result was not repeated during multiple experiments on STS-26... perhaps due to heating artifacts in the original experiment. Hence, although the viscosity dependencies suggest that the viscous dissipation in the continuous, wetting phase is a strong determinant we can as yet draw no firm conclusion regarding the dominant demixing mechanism operating." (4, p. 406)

It was also reported that "STS-26 results were somewhat compromised by small accelerations (evidenced by slow [mixing] ball movement...) the unexpected experimental temperature and lack of an expected photo showing the completely demixed state of the unit." (4, p. 406)

Key Words: Biotechnology, Phase Partitioning, Phase Separation, Separation of Components, Immiscible Fluids, Aqueous Solutions, Polymers, Two-Phase System, Dispersion, Liquid/Liquid Dispersion, Liquid/Liquid Interface, Drops, Drop Coalescence, Droplet Dispersion, Solid/Liquid Interface, Liquid Mixing, Liquid Demixing, Buoyancy Effects, Sedimentation, Contact Angle, Wetting, Particle Wetting, Wetting of Container, Non-Wetting of Container, Coated Surfaces, Container Shape, Crucible Effects, Aspect Ration, Viscosity, Biological Cells, Acceleration Effects, Thermal Environment More Extreme than Predicted, Contained Fluids, Liquid Crystals

Number of Samples: eighteen
Sample Materials: See above and Reference (4).
Container Materials: Machined Plexiglas™

Experiment/Material Applications:
See Brooks et al., STS-023

"The PPE systems flown on STS-26 [see Table 3, Reference (4)] were chosen to reproduce and augment... [STS-023] results and provide data concerning control of the demixing and final disposition of the phases." (4, p. 403)

References/Applicable Publications:
(1) Dumoulin, J.: Phase Partitioning Experiment on STS-26. NASA Fact Sheet, George C. Marshall Space Flight Center, June 1988. (preflight)

(2) "Seven Marshall Payloads to Fly on STS-26 in June. Marshall Star, Vol. 28, No. 5, October 7, 1987, pp. 1-2. (preflight; very short description)

(3) NASA Press Kit, Space Shuttle Mission, STS-26, September 1988, pp. 32-35. (preflight)

(4) Van Alstine, J. M., Bamberger, S., Harris, J. M, Snyder, R. S., Boyce, J. F., and Brooks, D. E.: Phase Partitioning Experiments on Shuttle Flight STS-26. In Seventh European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-295 (January 1990), pp. 399-407. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 11
Launch Date/Expt Date: April 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-5 containing six rotary seated helix fusion cells and one capillary chamber with temperature control and homogenizing device. (The equipment was newly developed for TEXUS 11.)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Electrofusion of Yeast Protoplasts

When two living organisms sexually unite, genetic characteristics of each combine to establish a new genetic code. While this is the most common method of genetic combination, electrically induced fusion of wall-less cells (protoplasts) also allows this exchange of genetic information. During the electrofusion process, repulsive forces of the cells are overcome by a weak, alternating electric field which is placed across the cell medium (dielectrophoresis). The field aligns the cells and brings the membranes of the fusion partners into close contact. After this is achieved, the a.c. field is switched off and a series of short duration, high intensity field pulses is applied to the cell medium. These pulses charge and break down (reversible break down) the membranes resulting in the fusion of the protoplasts.

This TEXUS 11 experiment was the first in a series of investigations designed by Zimmermann et al. to study the effect of the low-gravity environment on the electrofusion process. The specific objectives of the research were to (1) fuse protoplasts from two genetically different yeast strains and (2) determine if an improvement of the fusion process resulted under low-gravity conditions.

Six temperature-controlled, rotary seated helix chambers and one temperature-controlled capillary fusion chamber were used for the fusion experiments. The cell fusion in the capillary chamber was observed via a microscope lens and documented by a 90 frame camera. All of the chambers were equipped with a homogenizing unit to prevent settling of the biological material before and during launch.

During the flight, an inhomogeneous a.c. field (6 volts, 3 MHz) was applied to the biological material in each of the chambers. The cell-aligning dielectrophoresis process lasted 1 minute in three of the helix chambers and 2 minutes in the remaining helix chambers (2 minutes is the optimum dielectrophoresis time under 1-g conditions). Fusion was then initiated by switching off the a.c. field and applying a rectangular d.c. pulse. <Note: Details concerning the experiment sequence in the capillary chamber could not be located.>

Post-flight examination of the film documenting the fusion process in the capillary chamber indicated that only two protoplasts were located in the microscope's focused image plane. Thus, documentation of the process was not achieved. However, post-flight examination of the material within the helix chambers indicated a two-fold increase of hybrid yield over electrofusion experiments performed on Earth. The 2-minute time for dielectrophoresis resulted in a higher production of hybrid than the 1-minute time (this was also the case for 1-g experiments). It should be noted that the ground experiments did not utilize the same (1) protoplasts preparations as the low-gravity experiments or (2) dielectrophoresis shut-off times before and after the fusion pulse applications.

Key Words: Biotechnology, Electrofusion, Dielectrophoresis, Biological Cells, Protoplasts, Electric Field, Cell Fusion, Reversible Electric Breakdown, Sedimentation, Thermal Convection, Sample Homogeneity, Medical Applications, Contained Fluids, Photographic Difficulties

Number of Samples: seven experiment chambers
Sample Materials: yeast strain AH 22 pADH-040-2 HIS 4- X AH215 Leu 2- HIS3 (protoplasts)
Container Materials: helical chambers: Perspex^{TM}; capillary fusion chamber: unknown

Experiment/Material Applications:
The electrofusion process is employed to produce cell hybrids which can be applied in the biotechnological, medical, and agricultural sciences. This electrofusion technique has been applied successfully (high yields) to mammalian cells, bacteria, yeast protoplasts, etc. However, some types of hybrid generation require the use of a small number of fusion partners. Electrofu-

sion of these systems is difficult to achieve on Earth because (1) sedimentation prevents the necessary pearl-chaining process during the alignment phase and (2) thermal convection interferes with the required close contact of the cells. These gravity-induced phenomena can be overcome by the application of stronger electric fields. However, these fields tend to damage the fusion partners. The low-gravity environment would, theoretically, allow the fusion process to occur using a lower field strength that would be less damaging to cell structures.

References/Applicable Publications:

(1) Zimmermann, U., Büchner, K. H., and Schnettler, R.: Electrodiffusion of Yeast Protoplasts. In TEXUS 11/12 Abschlussbericht 1985, pp. 39-41. (post-flight; in German)

(2) Experiment Module TEM 06-5. In TEXUS 11/12 Abschlussbericht 1985, pp. 37-38. (post-flight; in German)

(3) Schnettler, R., Gessner, P., Zimmermann, U., Neil, G. A., Urnovitz, H. B., and Sammons, D. W.: Increased Efficiency of Mammalian Somatic Hybrid Production under Microgravity Conditions During Ballistic Rocket Flight. Applied Microgravity Technology, Vol. 2, February 1989, pp. A3-A9. (post-flight)

(4) Input received from Co-Investigator R. Schnettler, September 1989.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 13
Launch Date/Expt Date: April 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Cell TEM 06-5. The specific contents of the facility are unclear based on References (1) and (2). It appears that the temperature controlled container housed six helix chambers and either (1) a capillary fusion chamber or (2) a Plexiglas™ control vessel holding non-fused protoplast mixture. Reference (3) indicates that the module was improved post TEXUS 11 to allow movement of the capillary chamber in three-axes by telecommand.
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Federal Republic of Germany

Experiment:
Electrofusion of Yeast Protoplasts

This TEXUS 13 experiment was the second in a series of investigations designed by Zimmermann et al. to study the effect of the low-gravity environment on the electrofusion process (see Zimmermann, TEXUS 11). The specific objectives of the research were similar to those outlined under Zimmermann, TEXUS 11.

While Reference (1) indicates that the TEXUS 13 experimental setup was very similar to the TEXUS 11 setup, Reference (2) indicates that a somewhat different setup was used. For example, Reference (1) indicates that the fusion process took place in the TEXUS experiment module TEM 06-5 which held six temperature-controlled helix chambers and one capillary chamber. The cell fusion in the capillary chamber was observed via a microscope lens and documented by a television camera. The camera image was transmitted to the ground during flight.

In contrast, Reference (2) indicates that the fusion process took place in six chambers, but no mention is made of the capillary chamber. Instead, the reference details a Plexiglas™ control vessel which held the protoplast mixture but did not electrically fuse the material.

Although the field intensity and frequency of the current during dielectrophoresis was different from that of TEXUS 11 (here 300 V/cm, 2 MHz), the rest of the experimental fusion conditions were

similar to those employed on TEXUS 11. New ground-based hardware, which had not been available during the earlier TEXUS 11 flight, allowed identical, parallel, flight/ground experiments to be performed.

Post-flight, the protoplasts were placed on selection media so the cell wall could regenerate. However, a reversion of the genetic marker of one fusion partner resulted in "...a definite background growth of regenerating yeast protoplasts on the Petri dishes that were ready for evaluation." (1, p. 35 (translation)) Therefore, a count of the fusion products was impossible.

No further information concerning this experiment appears to be available at this time.

Key Words: Biotechnology, Electrofusion, Dielectrophoresis, Biological Cells, Protoplasts, Electric Field, Cell Fusion, Reversible Electric Breakdown, Sedimentation, Thermal Convection, Medical Applications, Contained Fluids, Deterioration of Samples After Low-G Flight

Number of Samples: Unclear; there appears to have been seven experiment chambers.
Sample Materials: yeast Strain AH 22 pADH-040-2 HIS 4- X AH215 Leu 2- HIS3 (protoplasts)
Container Materials: helical chambers: Perspex™; capillary chamber: unknown; control vessel: Plexiglas™

Experiment/Material Applications:
See Zimmermann, TEXUS 11.

References/Applicable Publications:
(1) Zimmermann, U., Büchner, K. H., and Schnettler, R.: Elektrofusion von Hefeprotoplasten. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 34-35. (post-flight; in German)

(2) Experiment-Modul TEM 06-5, In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 30. (post-flight; experimental apparatus description)

(3) Input received from Co-Investigator R. Schnettler, September 1989

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-11 containing nine helix chambers for the treatment of yeast cells
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Electrofusion of Yeast Protoplasts

This TEXUS 14a experiment was the third in a series of investigations designed by Zimmermann et al. to study the effect of the low-gravity environment on the electrofusion process (see Zimmermann, TEXUS 11, TEXUS 13).

The specific TEXUS 14a experiment goals and equipment setup were not detailed in the available publications.

Reportedly, due to an unexpected "wobbling motion" of the TEXUS rocket, uncontrollable accelerations were produced on the vehicle and the desired low-gravity level of 10^{-4} g was not attained. The experiment was flow again on TEXUS 14b (see Zimmermann, TEXUS 14b).

Further documentation of any TEXUS 14a results does not appear to be available.

Key Words: Biotechnology, Electrofusion, Biological Cells, Electric Field, Cell Fusion, Protoplasts, Medical Applications, Acceleration Effects, Rocket Motion, Contained Fluids

Number of Samples: Unknown; there appears to have been nine experiment cells.
Sample Materials: Unknown, possibly the same as those employed on TEXUS 11 and 13: yeast strain AH 22 pADH-040-2 HIS HIS 4- X AH215 Leu 2- HIS3 (protoplasts)

Container Materials: unknown, possibly Perspex™

Experiment/Material Applications:
See Zimmermann, TEXUS 11

References/Applicable Publications:
(1) Experiment-Modul TEM 06-11. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 97. (in German; experiment module)

(2) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 53-55. (in German; post-flight)

(3) Input received from Co-Investigator R. Schnettler, September 1989.

(4) Zimmermann, U., Büchner, K. H., and Schnettler, R.: Elektrofusion von Hefeprotoplasten. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 34-35. (post-flight; in German; appears to discuss 14b only)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 14b
Launch Date/Expt Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-11: Twelve helix chambers were employed to examine electrofusion and three additional chambers were employed to examine electric field induced gene-transfer. (TEM 06-11 was modified post TEXUS 14a to include additional helix chambers.)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
1. Electrofusion of Yeast Protoplasts
2. Electrofusion of Mammalian Cells
3. Electric Field Induced Gene Transfer

This TEXUS 14b experiment was the fourth in a series of investigations designed by Zimmermann et al. to study the effect of the low-gravity environment on the electrofusion process (see Zimmermann, TEXUS 11, TEXUS 13, TEXUS 14a). A detailed discussion of the electrofusion process can be found under Zimmermann, TEXUS 11.

While earlier TEXUS experiments by Zimmermann et al. studied the electrofusion of yeast protoplasts alone, this experiment also investigated (1) the electrofusion of mamalian cells and (2) the electric-field gene transfer (or electrically induced transfection) of mammalian cells. These two additional experiments were included to investigate the high-yield production of antibody-secreting hybridoma cells.

"In electrically-induced fusion, the cell membranes of the fusion partners are brought into close contact in a nonhomogeneous electrical alternating field (dielectrophoresis). The melting of the fusion partners is then triggered by short-duration voltage pulses (μsec) (reversible penetration of cell membranes). In electrical gene transfer, the reversible electrical penetration is used to permeabilize the cell membranes temporarily. In this way, one can infiltrate into the cells macromolecules (such as desoxyribonucleic acid) and the genetic information can be expressed." (2, p. 98 (translation)) Under low-gravity conditions it is possible that sedimentation and, more importantly,

thermally-induced convection (both gravity-induced effects) will not disturb the permeabilization of the cells.

All of the TEXUS 14b electrofusion and gene transfer experiments were performed in the TEXUS Experiment Module TEM 06-11. The module was constructed such that sample materials could be loaded just prior to the rocket launch if necessary. (Reportedly, the TEXUS 14b version of the module was an improved version of the earlier TEXUS 11 experimental unit used by Zimmermann.)

During the mission, the electrofusion of yeast protoplasts was performed in nine helix chambers; the electrofusion of mammalian cells was performed in three helix chambers, and the gene transfer of mammalian cells was performed in three transfection chambers. Helix chambers 1-3 contained "freshly prepared" yeast protoplasts, helix chambers 4-9 contained yeast protoplasts which had been stored 24 hours, helix chambers 10-12 contained SP2 cells and helix chambers 13-15 contained SP2 cells/plasmid pSV2-neo. A ground-based experimental module similarly processed cell preparations from the same protoplast and cell batches as had been placed in the rocket. <Note: Further details of the specific processing procedures for each of the helix chambers were presented in table form (see Reference (2), p. 100).>

1. Yeast Protoplasts Results

Post-flight analysis of the nine yeast protoplast chambers indicated that the absolute yield of yeast hybrids produced during the TEXUS 14b experiment was much higher than previous TEXUS experiments. This higher yield was attributed to "...the development of suitable preliminary incubation media, to keep the 'fusiogenity' of the cells constant for more than 24 hours." (2, p. 101 (translation)) Analysis showed, however, that it was not possible to determine the exact increase in relative hybrid yield under low-gravity conditions, possibly due to the following conditions:

"1. Variation in the appearance of multiple fusions which could not be investigated in this experiment could have led to statistical fluctuations in the hybrid yield.... Possibly, the relatively brief μg phase, considering the biological variability, is responsible for this.

"2. Analysis furthermore showed that temperature changes did appear in the chambers. The heating of the chambers was accomplished by virtue of the output introduction of the electrical fields that were applied and insufficient heat evacuation. Be-

sides, transition resistances on the sliding contacts presumably led to differences in the voltage supply of the chambers and thus to fluctuations in the yields within comparable fusion chambers.... With all due scientific caution, one can of course say that on the basis of these experiments that the hybrid yield was increased under μg conditions." (2, p. 102 (translation))

<Note: More detailed results of these electrofusion experiments were not presented.>

2. Mammalian Cell Results

Post-flight analysis of the mammalian cell electrofusion indicated that a large degree of multiple fusion had occurred when compared to the control (1-g) experiments. It was reported that the presence of multiple fusion could indicate that "...the melting process of the membranes (boundary surfaces) of the fusion partners is altered in a dielectrophoretically arranged cell." (2, p. 100 (translation)) When these results were compared to extensive post-flight ground experiments, the following conclusions were reached:

(1) "The hybrid yield decreases drastically in [the] case of longer storage time in weak conducting solutions. Because of this decline, flight and ground experiments must be performed simultaneously." (2, p. 100 (translation))

(2) "The appearance of multiple fusion could possibly be traced back indirectly to an intracellular ATP (and/or calcium level?) which was altered under μg conditions." (2, p. 101 (translation)) Changes in ATP and/or calcium can alter membrane fluidity and the mixing of the membrane components (fluidity and mixing are a function of gravity levels).

(3) "Under terrestrial conditions, the probability of the fusion of a cell pair within a chain is constant so that one can explain that, in spite of differing cell length, fusion takes place preferably between two (and, to a lesser extent, three) cells...." (2, p. 101 (translation)) Therefore, since multiple fusion is present, it must be assumed that this law is not valid under low-gravity conditions.

(4) In order to prove that low-gravity conditions influence the electrofusion process, "...the simultaneous investigation of the fusion products and fusion yield as well as the determination of the hybrid yield..." is required. (2, p. 101 (translation))

<Note: More detailed results of these electrofusion experiments were not presented.>

3. Gene Transfer Results:

Only a small number of samples were dedicated to the electrical transfection portion of the experiment. Although, for technical reasons, optimum conditions for this work could not be achieved, it was still shown that an increase in the yield of stable transformants (by one order of magnitude) could be realized. The yield from one chamber was equal to zero because of problems which arose during the transfer of electrical fields and during heat evacuation.

<Note: More detailed results of these gene transfer experiments were not presented.>

Key Words: Biotechnology, Electrofusion, Dielectrophoresis, Biological Cells, Protoplasts, Electric Field, Cell Fusion, Cell Storage, Electrotransfection, Reversible Electrical Breakdown, Electrical Gene Transfer, Sedimentation, Thermal Convection, Liquid Mixing, Medical Applications, Antibodies, Contained Fluids, Thermal Environment More Extreme Than Predicted, Deterioration of Loaded Samples Prior to Launch, Processing Difficulties

Number of Samples: fifteen experiment cells
Sample Materials: Yeast system: protoplasts of genetically marked strains of the Saccharomyces cerevisiae varieties; animal cells: hybridoma cell line and subcloned SP2 cells (Myeloma strain SP2 X G8 Hybrid Hybridoma Strain, Myeloma Strain Sp2 + pSV2-neo Plasmid). Helix chambers 1-3 contained yeast protoplasts, freshly prepared; Helix chambers 4-9, yeasts protoplasts stored 24 hours; Helix chambers 10-12, SP2 cells; Helix 13-15, SP2 cells/plasmid pSV2-neo. Cell Fusion Media: Yeast; 0.5 mM Mg-Acetate, 0.1 mM Ca-Acetate, 1.2 M Sorbite: SP2; 0.5 mM Mg-Acetate, 0.1 mM Ca-Acetate, 0.28 M Sorbite, 1 mg/ml Serum-Albumin. Gene Transfer Media: 220 mM Inosite, 30 mM KCl, 0.3 mM KH_2PO_4, 0.85 mM K_2HPO_4, pH 7.2.
Container Materials: unknown

Experiment/Material Applications:
See Zimmermann, TEXUS 11.

The hybridoma cell line and the subcloned SP2 cells "...differ from each other by virtue of the genetic markings but they reveal similar cell sizes.... The pSV2-neo plasmid leads to cells that are resistant against the antibiotic Neomycin." (2, p. 99 (translation))

References/Applicable Publications:

(1) Experiment-Modul TEM 06-11. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 97. (in German; experiment module)

(2) Zimmermann, U., Büchner, K. H., and Schnettler, R.: Zellfusion und elektrisch induzierter Gentransfer. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 98-102. (in German; postflight; appears to be 14b only)

(3) Input received from Co-Investigator R. Schnettler, September 1989.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 18
Launch Date/Expt Date: May 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Unknown, possibly the same as TEXUS 14b: TEM 06-11.
Builder of Processing Facility: Unknown, possibly the same as TEXUS 14b: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
1. Electrofusion of Yeast Protoplasts
2. Electrofusion of Mammalian Cells
3. Electric Field Induced Gene Transfer

This TEXUS 18 experiment was the fifth in a series of investigations designed by Zimmermann et al. to study the effect of the low-gravity environment on the electrofusion process (see Zimmermann, TEXUS 11, TEXUS 13, TEXUS 14a, TEXUS 14b). The specific objectives of the experiment were to examine (1) the electrofusion of yeast protoplasts, (2) the electrofusion of mammalian cells, and (3) electric field-induced gene transfer.

Electrofusion of Mammalian Cells

The fusion of the mammalian cells took place in six temperature-controlled, rotary-seated helical chambers: three containing low conductive medium (LCM) and three containing high conductive medium (HCM). "The fusion was performed during the low-gravity phase of the rocket's trajectory using programmed electrical field conditions as follows: dielectrophoretic alignment was performed by application of an alternating current with field strength of 250 V/cm at a frequency of 2 MHz applied for 30 seconds pre- and post-fusion pulse administration. The alignment field was switched off for 5 ms before and after each pulse to prevent current flow through the permeabilized [sic] cells.... Fusion was achieved by application of a total 3 pulses with a field strength of 2.25 kV/cm and a duration of 15 μs at 1 second intervals. The ground-based experiments were fused at the same time using an identical protocol." (1, p. 4)

Post-flight comparison of the ground-based and flight-based mammalian cell fusion experiments indicated that the reduced gravity hybrid yields were greater by a factor slightly greater than 2 over the 1-g experiments. This was true for both the low and high conducting mediums. It was also evident that the efficiency of hybrid generation was greater for the HCM (for both 1-g and low-gravity conditions). Analysis of "control" chambers onboard the sounding rocket which were not exposed to the electric fields indicated that "...the frequency of spontaneous fusion in this system was zero." (1, p. 7)

Because the cells had been incubated in their respective media (a) 3.0 hours prior to when the fusion actually took place (during launch operations) and (b) 1.5 hours after fusion was complete (during rocket recovery operations), subsequent ground-based experiments were performed to determine the effect of the extended incubation time on the cell viability and hybrid yields. It was found that after 3 hours of incubation at 23 ^{o}C "...15-20% of the cells incubated in HCM and 25-30% of the cells incubated in LCM were dead." (1, p. 7) Reportedly, the HCM was comprised of the LCM supplemented with 4.0 mM potassium acetate. As expected, the addition of potassium acetate (HCM) increased the survivability of the cells.

Electrofusion of Yeast Protoplasts/Electric Field-Induced Gene Transfer

Because of the malfunction of some helical chambers, no results were obtained from the yeast protoplast electrofusion experiments. Results from the electric field-induced gene transfer investigation were reported to have been similar to those of the TEXUS 14b mission (see Zimmermann, TEXUS 14b). <Note: No specific discussion of the TEXUS 18 gene transfer results could be found.>

Key Words: Biotechnology, Electrofusion, Dielectrophoresis, Biological Cells, Protoplasts, Electric Field, Cell Fusion, Cell Storage, Incubator, Cell Survivability, Gene Transfer, Medical Applications, Contained Fluids, Hardware Malfunction, Deterioration of Loaded Samples Prior to Launch, Deterioration of Samples After Low-G Flight

Number of Samples: See Experiment Section; protoplasts

Sample Materials: Yeast strains were unspecified.
Mammalian strains were G8 hybridoma and SP2/0-UZ hybridoma clone.
Cell fusion media- (1) LCM: 0.28 M sorbitol, 0.5 mM Mg-acetate and 0.1 mM Ca-acetate, 1 mg/ml bovine serum albumin (Serva); (2) HCM: LCM supplemented with 4.0 mM potassium acetate. The conductivity (at 25 ^{o}C) of the LCM was 100 μS/cm, and of the HCM was 415 μS/cm.
Container Materials: unknown

Experiment/Material Applications:
These specific mammalian fusion partners were selected because the hybrids formed by these partners are resistant to a hypoxanthine-aminopterin-thymidine (HAT) supplemented medium.

See also Zimmermann, TEXUS 11.

References/Applicable Publications:
(1) Schnettler, R., Gessner, P., Zimmermann, U., Neil, G. A., Urnovitz, H. B., and Sammons, D. W.: Increased Efficiency of Mammalian Somatic Cell Hybrid Production under Microgravity Conditions During Ballistic Rocket Flight. Applied Microgravity Technology, Vol. 2, February 1989, pp. A3-A9. (post-flight)

(2) Input received from Co-Investigator R. Schnettler, September 1989.

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Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Columbia)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck, Two STS Middeck Lockers
Processing Facility: Storage facility to hold human red blood cells, white cells and platelets under blood bank conditions
Builder of Processing Facility: Arthur D. Little. Inc., Cambridge, Massachusetts

Experiment:
Initial Blood Storage Experiment (IBSE)

The specific objective of this STS middeck experiment was to investigate the behavior of human red blood cells, white cells, and platelets during 6 days and 2 hours of exposure to the STS low-gravity environment.

During the mission, two Initial Blood Storage Experiment (IBSE) modules were housed in the shuttle's middeck area. The specially designed and fabricated electrically-powered hardware units provided appropriate environmental temperatures and air flows to support cell metabolism throughout the experiment. "The plastic bags containing the blood cells were placed on racks within closed, temperature controlled stainless steel Dewar chambers. A continuous flow of air through the chambers and around the bags was provided to support cell respiration and remove carbon dioxide during the experiment. Red cells and white cells were held at 5 ^{o}C (...[+/-] 1) and platelets at 22 ^{o} C (...[+/-] 1)." (3, p. 606) Identically controlled sets of blood cells were contained on Earth for comparison.

Reportedly, "The blood samples were stored in the orbiter on the launch pad for approximately 12 h before lift-off. Launch subjected the blood samples on the orbiter to an acceleration of 2.9 g's for a period of 3 min. The investigators determined that 3 min of the increased gravity would not result in any significant, additional sedimentation of the cells. The samples stored on Earth, therefore, were not subjected to an identical launch acceleration profile. Also, the bags containing platelets were specifically oriented in the IBSE hardware so that the prelaunch

and launch accelerations were in the long direction of the bags. This orientation minimized the number of cells which would come in contact with the "lower" boundaries of the bag during the period of increased acceleration.

"Launch also subjected the mid-deck lockers in the orbiter to a vibration environment that was not experienced by the lockers stored on Earth. The IBSE module was designed to float in a block of Pyrell... foam that fully filled the space between the module and the interior walls of the locker. The launch vibrations felt by the IBSE hardware were, therefore, significantly attenuated. The random vibration level during launch begins at 20 Hz and is at its highest levels of 0.04 g^2/Hz between 150 and 1000 Hz. The first mode, isolated frequency of the IBSE, within the foam, in any direction, was approximately 10 Hz. The transmissibility at 150 Hz is about 0.1. As a result, the vibration levels experienced by the IBSE spaceflight hardware and blood samples were extremely low. No additional effort was expended to either reduce further the vibration levels experienced by the blood samples on board the orbiter or to subject the blood samples stored on Earth to a similar transient, vibration environment." (1)

Samples within the STS IBSE modules were carefully compared to identically controlled sets of blood cells contained on the ground. To eliminate bias, blind assignment was used to allocate blood cell samples to the low-g and 1-g experiments. To assure blind assessment of the experimental findings, post-experiment samples for measurement were identified only in code. To optimize the chances of detecting possible gravitational effects, a wide array of measurements of cellular, function, morphology, metabolism, and immunology were made. Analysis of variance was used in analyzing the data.

Reportedly, "The most striking result was the finding that platelets displayed markedly superior structural and functional integrity at microgravity. Granulocytes held on the ground were preserved slightly better than those that orbited in the shuttle, whereas red cells displayed few effects that were attributable to the gravitational variable. Polyvinylchloride-di-(2-ethylhexyl)phthalate (PVC-DEHP) was the plastic of choice for the storage of red cells, while PVC-trioctyltrimellitate (PVC-TOTM) was superior to PVC-DEHP and polyolefin (PO) for platelets." (3, p. 605)

Additional information concerning this experiment can be found in Reference (3).

Key Words: Biotechnology, Biological Cells, Cell Storage, Cell Survivability, Metabolism, Vibration Isolation Systems, Fluid Motion Damping, Acceleration Effects, Sedimentation, Contained Fluids, Refrigeration, Medical Applications

Number of Samples: 28 sealed bags
Sample Materials: 9 bags of Human red blood cells (erythrocytes, 250 mL each); 9 bags of white cells (leukocytes, 75 mL each) suspended in plasma phosphate-buffered saline (PBS) glucose; and 10 bags of platelet concentrate (60 mL each)
Container Materials: Bags made of three different polymer/plasticized formulations: (1) Polyvinylchloride plasticized with di-(2-ethylhexyl)phthalate (PVC-DEHP), (2) PVC plasticized with trioctyltrimellitate (PVC-TOTM), and (3) unplasticized polyolefin.

Experiment/Material Applications:
"A specific aim of the investigation was to improve the understanding of basic blood cell physiology while contributing to an improvement in the survival and efficacy of blood cells for transfusion." (1, p. 1215) The research was also performed to help plan for emergency medical support for astronauts during prolonged space flight.

References/Applicable Publications:
(1) Almgren, D. W., Csigi, K. I., Glaser, P. E., Lucas, R. M., and Spencer, R. H.: The Initial Blood Storage Experiment-The Spaceflight Hardware Program. In Aviation, Space, and Environmental Medicine, December 1989, pp. 1215-1221. (hardware setup; some post-flight results)

(2) Input received from Experiment Investigator, August 1989 and July 1993.

(3) Surgenor, D. M., Kevy, S. V., Chao, F. C., Lionetti, F. J., Kenney, D. M., Jacobson, M. S., Kim, B., Ausprunk, D. H., Szymanski, I. O., Button, L. N., Curby, W. A., Luscinskas, F. W., Curran, T. G., Carter, J. H., Carr, E., Mozill, D. R., Blevins, D. K., and Laird, N.: Human Blood Cells at Microgravity - The NASA Initial Blood Storage Experiment. Transfusion 1990; 30:605-616.

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Principal Investigator(s): Hampp, R. (1)
Co-Investigator(s): None
Affiliation(s): (1) Universität Tübingen, Germany

Experiment Origin: Federal Republic of Germany
Mission: TEXUS 17
Launch Date/Expt. Date: May 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-5 (Electrofusion facility including microscope optics, video downlink, and telecontrol)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Germany

Experiment:
Electrofusion of Plant Cell Protoplasts

This TEXUS 17 experiment was designed to study the low-gravity electrofusion of vacuolated mesophyll protoplasts with evacuolated mesophyll protoplasts.

The specific objective of the investigation was to determine if the hybrid yield of the protoplasts could be increased when the detrimental effects of gravity-induced convection and sedimentation were reduced. <Note: A detailed description of the principles of electrofusion can be found under Zimmerman, TEXUS 11 (this chapter).>

The investigation was performed in the TEXUS Experiment Module TEM 06-5 electrofusion facility. (This facility was the same as that used by Zimmerman during the TEXUS 11 mission. See Zimmerman, TEXUS 11 for a description of the hardware.) The apparatus allowed viewing of the protoplasts (100-200x total magnification) throughout the experimental procedure.

During the mission, the electrofusion chamber was filled and Nicotiana tabacum protoplasts were aligned using a weak alternating field (2 MHz; 160 V/cm, peak to peak). When short chains of protoplasts were visible, a fusion pulse was applied (0.9 kV/cm; 50 μs). It was possible, through telecontrol, to direct various real-time, critical steps such as pump activation, flow direction, and pulse application. Other portions of the procedure were automatic. (Reference (2) contains detailed descriptions of the preflight material preparation procedures, experimental procedure, and post-flight sample handling procedures.)

Post-flight microscopic analysis of the sample material revealed a significant increase in hybrid cells (homo- and heterospecific)

over corresponding 1-g experiments. "Evaluation of about 1000 protoplasts (vacuolated and evacuolated) yielded about 120 clearly distinguishable 1:1 hybrids, i.e. about 12% of all cells submitted to the fusion procedure. This is about 10 to 15 times more compared to fusion under terrestrial conditions and, in real numbers, about 0.5 x 10^6 heterospecific 1:1 hybrids out of 4 x 10^6 protoplasts introduced into the fusion chamber. This yield is sufficiently high to be used for biochemical analysis or characterization of the metabolic state...." (2, p. 1176)

<Note: Reference (4) (as listed below) was not available to aid in the preparation of this experiment summary.>

Key Words: Biotechnology, Electrofusion, Dielectrophoresis, Electric Field, Cell Fusion, Biological Cells, Protoplasts, Hybrid Viability, Metabolism, Liquid Transfer, Convection, Sedimentation, Contained Fluids, Medical Applications

Number of Samples: One experiment run on 4 x 10^6 protoplasts
Sample Materials: Evacuolated (without a cavity) and vacuolated (with a cavity) mesophyll protoplasts of Nicotiana tabacum
Container Materials: Fusion chamber: Perspex™ (Plexiglas™)

Experiment/Material Applications:
The selected protoplasts are a model system for fusion/hybrid culture and regeneration, and metabolic responses.

References/Applicable Publications:
(1) Hampp, R., Mehrle, W., and Naton, B: Elektrofusion bon Pflanzenprotoplasten. In TEXUS 13-16 Abschlussbericht, 1988, pp. 30-33.

(2) Mehrle, W., Hampp, R., Naton, B., and Grothe, D.: Effects of Microgravitation on Electrofusion of Plant Cell Protoplasts. Plant Physiol. Vol. 89, 1989, pp. 1172-1177.

(3) Input received from Experiment Investigator, October 1989 and June 1993.

(4) Hampp, R., Naton, B., Hoffmann, E., Mehrle, W., Schönherr, K., and Hemmersbach-Krause, R.: Hybrid Formation and Metabolism of Plant Cell Protoplasts Under Microgravity. The Physiologist, Vol. 35, Suppl., 1992, pp. S-27 - S-30.

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Co-Investigator(s): None
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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Microtubule Assembly

"Chameleon skin changes color by moving small, black granules (melanosomes) from inside the epidermis of the skin to outside. This process is predictable and can be initiated by chemicals (MSH and Norepinephrine) as well as by light. These melanosomes are guided and prodded in their movement by filaments which... [also] help to maintain the three dimensional architecture of the cell. The filaments (microtubules and microfilaments) assemble and turnover rapidly; they are the structural elements which keep organelles from sedimenting to the bottom of the cell and also maintain the inside to outside relationship. Since color changes are rapid (<2 min) and the skin is hearty, this model skin is ideally suited to short term... [sounding] rocket experiments." (5)

This Consort 1 experiment was designed to determine if cell secretion of important molecules is altered in a low-gravity environment. "In order to examine the assembly and function of microtubules in microgravity... [a series of experiments have been developed] the first of which was aboard Consort I, that investigates the hormonally induced intracellular transport of pigment granules within the pigment cells of the chameleon." (6, p. 30)

The materials used in this experiment were chosen to match the restraints of the Consort 1 rocket and the MDA. "Specifically, a cell type (able to be stored for 24-30h before launch) exhibiting a microtubule dependent, hormonally stimulated response within 2-3 min was needed. The cell type chosen was chameleon skin, suspended in amphibian Ringer's solution, and stimulated with

melanocyte stimulating hormone (MSH)." (4, p. 1))

The goals of the experiment included determining "(a) how long... the [chameleon] skin [can] be stored and still retain a hormone response; (b) how long... the melanocyte stimulating hormone (MSH) [can] be stored; (c) [if]... skin can be fixed by diffusion-driven processes (without active injection of solutions); and (d) [if] the skin respond[s] in microgravity." (4, p. 2)

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1).

This experiment was allotted 17 "Type 3" test wells. Each "Type 3" well provided the investigator with one data point. The well-type used three sample wells. <Note: it appears that one of these sample wells was in the top block and two of the sample wells were in the bottom block.> Reportedly, the wells in the bottom block of each well type were filled prior to flight with either (1) Ringer's amphibian buffer (RAB) ("...pink in color due to the pH 7.2 effect on the indicator dye (phenol red") (4, p. 3)), (2) melanocyte stimulating hormone (MSH) and RAB (pink in color), or (3) glutaraldehyde (clear).

Thirty minutes prior to loading the upper block, the flank skin was removed from chameleon lizards. The skin was then floated on RAB in a petri dish until it could be cut into small-sized pieces (15 mm). Green sections of skin were loaded into the upper wells. These upper wells were then "...filled with RAB taking care to expel all air." (4, p. 4) The blocks were joined together such that the wells in the upper block were purposely misaligned with the wells in the lower block. This prevented materials within the wells from coming into contact prior to the initiation of the experiment.

Once the rocket had been launched and the low-gravity phase had been achieved, a motor and cam mechanism moved the upper block to the right, aligning the skin/RAB wells with one of the three solutions contained in the bottom wells. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity phase, the upper block again moved to the right, aligning the well (which originally contained only the skin/RAB mixture) with another well on the lower block (which contained glutaraldehyde to stop the reaction). The fluids in these wells remained in contact throughout the re-entry period of the rocket.

Three ground-based control experiments were performed for comparative purposes. One ground experiment was performed at nearly the same time as the flight experiment and used chameleon skin tissue from the "same pool." The other two ground experiments were performed to investigate how long the skin tissue could be held and still retain MSH responsiveness. "The first was started about 12-h before the true control (to test for an extended tissue hold if the rocket launch were [sic] to be postponed) while the second was started 16 h after the true control (to test for responsiveness in a much fresher tissue)." (4, p. 4)

<Editor's Note: Presumably the "true control" was the ground-based control experiment which was performed at nearly the same time as the Consort 1 flight.>

The procedure for these ground-control experiments was somewhat different than the actual Consort 1 flight (see 4, p. 5).

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 3" test wells.

Examination of the contents of each well associated with the Consort experiment was initiated about 6 hours after the flight. Lengthy descriptions of the flight and ground-control wells were presented in Reference (4). The following comments/conclusions were made:

"(a) About three-quarters [of] the wells used were compromised by leakage or contamination problems.

"(b) This resulted in a limited [number of] replicates in each treatment group, to the point where no absolute statements can be made regarding responsiveness of the tissues in microgravity.

"(c) Great care should be taken in assigning neighbors in the MDA. If this is done it would minimize the damages caused by gratuitous donation of unwanted fluids....

"(e) Ground based control experiments established that:

"(1) Chameleons killed at T-36 h retained their green color but had a very muted response to MSH stimulation.

"(2) MSH prepared at T-48 h and tested at a 500-fold level above that needed for minimal levels of MSH stimulation adequately stimulated color change at the time of flight. Experiments are under way to establish the kinetics of MSH decay when stored in amphibian Ringer's solution.

"(3) Chameleons killed at T-20 h retained their green color and had a good response to MSH stimulation.

"(4) Chameleons killed at T-12 h also retained their green color and response....

"(f) Flight experiments can be summarized as follows:

"(1) Tissues that were moved at the start of microgravity so that they were situated over an equal volume of 30% glutaraldehyde and held there for the six minute flight were still green and contained sufficient (0.6-5%) glutaraldehyde by the end of the period so that fixation should have been achieved. The geometry of the chamber is such that the important unknown is where the tissue was relative to the interface, as highest fixa-

tive concentrations at the earliest times would have been achieved at the interface. Because the MDA was installed so that the g-force of spin vector was oriented to move tissues away from the interface, it is likely that the tissues were at the peripheral end of the chamber. Some microscopic characterization has been done, with the interpretation difficult. The tissue was still intact after launch and at the point of entry into microgravity. Many morphological details look ok, but doubts remain about the others.

"(2) Tissues that were moved at the start of microgravity so that they were situated over an equal volume of amphibian Ringer's solution (no MSH), held there for six minutes and then moved to an equal volume of 20% glutaraldehyde were recovered as a mixture of green and brown. Retention of color (green) would have been expected. Reasons for color change could include [1] donation of an unknown agent from adjacent wells to... [the wells used in this experiment] that caused the color change; [2] microgravity experience results in a movement of melanosomes in the tissue that causes color change independent of MSH; or [3] very high glutaraldehyde concentrations cause artifacts with regard to color changes. Ongoing microscopy studies of these samples and ground controls may help with the interpretation.

"(3) Tissues that were moved at the start of microgravity so that they were situated over an equal volume of amphibian Ringer's solution plus a 500-fold concentration of MSH, held there for six minutes and then moved to an equal volume of 30% glutaraldehyde were recovered as a mixture of green and brown (no different from the MSH-treated group). They were expected to be green (if they had not lost their responsiveness to MSH) or brown (if sufficient MSH was present to change them in the six minute period)...." (4, pp. 2-3)

Key Words: Biotechnology, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Solid/Liquid Interface, Cellular Characteristics, Cell Storage, Mass Transfer, Suspension of Particles, Chameleon Skin, Biological Cells, Cell Secretion, Salt Solution, Sedimentation, Contamination Source, Liquid Leakage, Contained Fluids, Medical Applications

Number of Samples: seventeen

Sample Materials: Top wells: chameleon skin and amphibian Ringer's salt solution (also called Ringer's amphibian buffer (RAB)); bottom wells: either (1) RAB, (2) RAB plus melanocyte stimulating hormone (MSH), or (3) glutaraldehyde
Container Materials: inert material

Experiment/Material Applications:
This investigation has commercial applications in the area of secretion research. "...optimizing cell secretion is a major concern of individuals and biotechnology companies involved in genetic engineering as well as those studying the relationship of cell secretion to human health and medicine." (6, p. 30) (See also the experiment summary above.)

"...the intracellular events involving microtubules in these particular [chameleon] cells are nothing short of dramatic.... [T]his system is particularly hardy and can withstand the timing and environmental conditions currently in the Consort mission protocol." (6, p. 30)

"Extrapolations of this model [system] include hormone secretion (inside to outside), intracellular transport (elimination of waste products) and the internal organization and architecture of cells." (5)

References/Applicable Publications:
(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Málaga, Spain, IAF #89-439. (post-flight)

(4) Hammerstedt, R.: Preliminary Report, Consort 1 Flight, PSU-Center for Cell Research, May 15, 1989. A report sent from R. Hammerstedt to W. Hymer, A. Mastro, S. Stein, J. Cassanto, P. Todd, E. Knuze, and L. Epp, May 21, 1989. (provided by ITA to C. A. Winter 1/91) (post-flight)

(5) Input received from Experiment Investigator, June 1991.

(6) Mount Union College News Release, 1989 (received from experiment investigator).

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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Collagen Polymerization

"A variety of different biological structures are formed by the directed assembly of large homogeneous protein macromolecules. Many of these processes have been simulated successfully in vitro. Yet, the products of such simulations often lack the specificity and material characteristics of the structures formed within biological systems....

"Presumably, the mismatch between in vivo and in vitro products derives, in part, from biophysical differences in the assembly environments. Whereas in the in vivo assembly occurs in viscous template-directed fashion, the in vitro assembly occurs in an environment with large scale bulk fluid motions, density driven flows and shear flows. The latter assembly conditions should foster disorder during macromolecule assembly unless template constraints are very stringent." (1, p. 1)

This experiment (together with those of Stodieck, Consort 1 and related KC-135 experiments) "...focuses upon using reduced gravity environments to evaluate in vitro macromolecule assembly processes and products. The experiments were designed to evaluate the effects of the reduced gravity assembly conditions on rates of assembly and product characteristics." (1, p. 1) More specifically, this Consort 1 polymerization experiment was designed to study the low-gravity formation of collagen structures.

"Collagen... is an important protein because it is polymerized into fibers that underlie the structural properties of nearly all body tissues." (3, p. 4) During the formation of collagen in the low-gravity environment, bulk fluid convective mixing, shear forces, and material sedimentation should be reduced. Such a reduction of these gravity-induced phenomena should result in "...the slower more homogeneous formation of biomaterials." (4, viewgraphs)

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2 and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two Teflon^{TM} blocks. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto et al., Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted two "Type 3" test wells. Discussion of this specific well-type is detailed here.

Each "Type 3" test well provided the investigator with one experimental opportunity. The well-type used three sample wells, two in the top block and one in the bottom block. Thirty-six hours prior to the rocket launch, the well in the lower block of each well-type was filled with solubilized collagen. Twelve hours later, one upper well of each well-type was filled with initiation buffer and the second upper well of each well-type was filled with 2% glutaraldehyde fixative. The long delay in the filling of the upper and lower wells "...was primarily due to the relatively large number of wells that had to be precisely loaded." (1, p. 3)

Reportedly, the fluids used in the investigation were chosen to match the opportunities/restraints of the Consort 1 rocket and the MDA. "Early" sample loading of the MDA (24-48 hours prior to launch), processing temperature of the samples (approximately 22 ^{o}C), high g loads experienced by the vehicle during takeoff and landing (up to 20g's), 10^{-5}-10^{-6}g low-gravity duration (approximately 7 minutes), and "late" retrieval of samples (approximately 4 hours post-flight) limited fluid selection and investigative objectives.

Prior to the rocket flight, the blocks were joined together such that the wells in the upper block were purposely misaligned with the wells in the lower block. This prevented materials within the wells from coming into contact prior to the initiation of the experiment. Once the rocket had been launched and the low-gravity phase had been achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells containing liquid solubilized collagen and the initiation buffer. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity phase, the upper block again moved to the right, aligning the well which originally contained only solubilized collagen (but now contained a mixture of solubilized collagen and buffer) with the well containing the liquid glutaraldehyde fixative. These wells remained in contact throughout the re-entry period of the rocket.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 3" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Post-flight analysis of the contents of the wells was performed. Reportedly, "Because of steep concentration gradients across the interface of the precursor and initiator solutions, diffusion was

expected to occur at relatively high rates. High rates of diffusion were essential to maximize the amount of material polymerized fibers to remain in the precursor well since fibers would exhibit very high molecular weights and thus would not diffuse away from the assembly location.

"As expected, plugs of collagen... materials... were localized to the well originally containing the precursor solutions. Plugs extended to approximately half of the 16-mm depth suggesting that this was the depth reached by diffusion initiator components. The plugs of material most importantly, were of sufficient size and quality to permit subsequent analysis steps.... Since the plugs have depths that are quite large compared to fiber lengths, an important series of questions can be addressed. These questions relate to the diffusion distances supported by these periods of high quality low gravity and to any intrinsic effect that diffusion may have on the polymerization process in these sensitive reactions." (3, p. 6)

Gross observations of the opacity of the collagen indicated that significant assembly of the three-dimensional lattice had occurred (the assembly did not appear to be complete).

It was further noted that:
(1) mixing was dependent upon small convective forces induced by positioning reactants adjacent to each other

(2) diffusion accounted for the remaining interfacial mixing

(3) the collagen materials, so formed vary from those prepared in ground control experiments.

No further information discussing the experimental results could be located at this time.

Key Words: Biotechnology, Medical Applications, Protein Macromolecules, Macromolecular Assembly, Collagen Structures, Biological Structures, Reactant Solutions, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Convection, Mass Transfer, Shear Forces, Fibers, Interface Physics, Sedimentation, Polymerization, Concentration Distribution, Contamination Source, Liquid Leakage, Contained Fluids

Number of Samples two
Sample Materials: Top wells: (a) collagen initiation buffer, (b) 2% glutaraldehyde fixative; bottom wells: solubilized collagen
Container Materials: Teflon™

Experiment/Material Applications:
"Materials formed from collagen precursors could have significant applications in medicine. This research will lead to a greater understanding of the effects of gravity on bulk fluid biomaterials processing and may enable the identification of processes that will allow the routine production of high quality bioproducts." (3, p. 4)

This investigation had commercial applications in the area of biofilms which are used for artificial skin implants, blood vessels, tendons, corneas, and other organs.

References/Applicable Publications:
(1) Moos, P. J., Hayes, J. W., Stodieck, L. S., and Luttges, M. W.: Macromolecular Assemblies in Reduced Gravity Environments. AIAA 28th Aerospace Sciences Meeting, January 8-11, 1990, Reno, Nevada, AIAA 90-0027, 8 pp. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results - A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(5) Input received from Principal Investigator M. Luttges, December 1991.

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Principal Investigator(s): Pellegrino, J. (1)
Co-Investigator(s): Unknown
Affiliation(s): (1) National Institute of Standards and Technology, Boulder, Colorado

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Chitosan Membrane Formation

"Thin film membranes cast on earth are subjected to convection forces which are believed to accentuate nonuniformity and variations in both porosity and macro molecular aggregation." (1, viewgraphs) In a low gravity environment, buoyancy-driven convection should be reduced and more uniform membranes are expected to form. Low-gravity experimentation could help to determine "...how gravity effects the formation of macromolecular aggregates during a film forming process." (1, viewgraphs)

The objective of this Consort 1 experiment was to study the role of convection in evaporative film formation.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (two other experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments including this one) and (b) determine the effect of reentry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the ap-

propriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for this and other film formation and membrane casting experiments. This experiment was allotted five "Type 4" test wells. Discussion of this specific well-type is detailed here.

Each "Type 4" test well provided the investigator with one experimental opportunity. The well-type used four sample wells, one on the top block and three on the bottom block. References (1) and (5) appear to differ on the specific materials which were placed in each well for this experiment. It is suspected that (a) the well in the top block of each well-type contained the membrane forming surface (either glass or a ceramic disc), (b) the first well in the bottom block of each well-type contained a solution of Polyglucosamin acetate, 2% in water, (c) the second well in the bottom block of each well-type contained either air or NaOH, 2% in water, and (d) the third well in the bottom block of each well-type contained either water or air.

Prior to flight, the blocks were joined together such that the well in the upper block was aligned with the first well in the bottom block. Thus, prior and during launch, the solution was in contact with the film-forming surface. Once the rocket had achieved the low-gravity phase, a motor and cam mechanism moved the upper block to the right, aligning the well on the upper block with the well on the lower block which contained air/NaOH. Although it was not specifically stated in the available publications, the air most likely dried the polymer solution forming the membrane. Just prior to the termination of the low-gravity phase of the rocket, the upper block again moved to the right, now aligning the well on the top block with the well on the bottom block which contained water/air. The blocks remained in this position throughout the re-entry period.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected and membranes were cast in the "Type 4" cells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Post-flight evaluation of the sample wells was initiated. "Membranes of chitosan and perfluorosulfonic acid (Nafion) [The quote is also referring to another of Pellegrino's Consort experiments (see Pellegrino, Consort 1, "Nafion Membrane Formation")] and composites of these two polymers were cast on 3-mm ceramic discs and examined by scanning electronic microscopy. These are currently undergoing comparison with their counterparts cast in the presence of gravity." (3, p. 7)

No further information discussing specific experiment objectives or results could be located at this time.

Key Words: Biotechnology, Membranes, Thin Film Membranes, Polymer Membranes, Convection, Porosity, Macromolecular Aggregates, Buoyancy Effects Diminished, Evaporation, Mass Transfer, Medical Applications, Pharmaceutical Applications, Liquid Leakage, Contamination Source

Number of Samples five
Sample Materials: The top and bottom well allocations as stated in References (1) and (5) and inferred in (3), appear to differ. Presumably-top wells: either glass or ceramic discs; bottom wells: (1) polyglucosamin acetate, 2% in acetone and water, (2) NaOH, 2% in water, (3) air
(Na*O*H*)
Container Materials: inert material

Experiment/Material Applications:
"Potential future commercial applications are: medical, pharmaceutical, industrial and scientific industries that have a need for filtration, purification technology, separation of... fluids, etc." (1, viewgraphs)

No specific application of the Chitosan was detailed.

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight; brief discussion of results)

(4) Letter from J. J. Pellegrino to J. M. Cassanto (ITA). April 12, 1989, describing "Quick Look at CONSORT 1 MDA Results-NIST Membrane Formation." (post-flight; information sent from ITA to C. A. Winter, NASA)

(5) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. Winter, 1/91)

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Co-Investigator(s): Unknown
Affiliation(s): (1,3,4) National Institute of Standards and Technology, Boulder, Colorado; (2) During Consort 1: National Institute for Standards and Techology (NIST), Boulder, Colorado, Currently: University of Colorado Department of Chemical Engineering, Boulder, Colorado

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Ceramic Membrane Casting

"Thin film membranes cast on earth are subjected to convection forces which are believed to accentuate nonuniformity and variations in both porosity and macro molecular aggregation." (1, viewgraphs) In a low-gravity environment, buoyancy-driven convection should be reduced and more uniform membranes are expected to form. Low-gravity experimentation could help to determine "...how gravity effects the formation of macromolecular aggregates during a film forming process." (1, viewgraphs)

The objective of this Consort 1 experiment was to study the role of convection in evaporative film formation.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (two other experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments including this one) and (b) determine the effect of reentry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for this and other film formation and membrane casting experiments. This experiment was allotted five "Type 4" test wells. Discussion of this specific well-type is detailed here.

Each "Type 4" test well provided the investigator with one experimental opportunity. The well-type used four sample wells, one on the top block and three on the bottom block. References (1) and (5) appear to differ on the specific materials which were placed in each well. It is suspected that (a) the well in the top block of each well-type contained the membrane-forming surface (ceramic disc), (b) the first well in the bottom block of each well-type contained a solution (A100H (boehmite) and water), (c) the second well in the bottom block of each well-type contained either air or some unspecified material, and (d) the third well in the bottom block of each well-type contained either air or water.

Prior to flight, the blocks were joined together such that the well in the upper block was aligned with the first well in the bottom block. Thus, prior and during launch, the solution was in contact with the film forming surface. Once the rocket had achieved the low-gravity phase, a motor and cam mechanism moved the upper block to the right, aligning the well on the upper block with the well on the lower block which contained air/unspecified material. Although it was not specifically stated in the available publications, the air most likely dried the polymer solution forming the membrane. Just prior to the termination of the low-gravity phase of the rocket, the upper block again moved to the right, now aligning the well on the top

block with the well on the bottom block which contained water/air. The blocks remained in this position throughout the re-entry period.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected and membranes were cast in the "Type 4" cells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Reportedly, "Permeation studies of 3-mm ceramic membranes cast during the flight are... underway; these membranes consist of a thin beohmite [sic] gel that is fired after casting." (3, p. 7)

No further information discussing specific experiment objectives, or results could be located at this time.

Key Words: Biotechnology, Membranes, Thin Film Membranes, Polymer Membranes, Convection, Porosity, Macromolecular Aggregates, Buoyancy Effects Diminished, Evaporation, Mass Transfer, Medical Applications, Pharmaceutical Applications, Liquid Leakage, Contamination Source

Number of Samples: five
Sample Materials: The top and bottom well allocations as presented in References (1) and (5) appear to differ. Presumably-top wells: ceramic disks; bottom wells: (1) A100H (boehmite) and water, (2) unspecified, (3) air (see also above experiment summary)
Container Materials: inert material

Experiment/Material Applications:
"Potential future commercial applications are: medical, pharmaceutical, industrial and scientific industries that have a need for filtration, purification technology, separation of... fluids, etc." (1, viewgraphs)

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight, brief discussion of results)

(4) Letter from J. J. Pellegrino to J. M. Cassanto (ITA). April 12, 1989, describing "Quick Look at CONSORT 1 MDA Results-NIST Membrane Formation." (post-flight; information sent from ITA to C. A. Winter, NASA)

(5) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. Winter, 1/91)

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Co-Investigator(s): Unknown
Affiliation(s): (1) National Institute of Standards and Technology, Boulder, Colorado

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Nafion Membrane Formation

"Thin film membranes cast on earth are subjected to convection forces which are believed to accentuate nonuniformity and variations in both porosity and macro molecular aggregation." (1, viewgraphs) In a low-gravity environment, buoyancy-driven convection should be reduced and more uniform membranes are expected form. Low-gravity experimentation could help to determine "...how gravity effects the formation of macromolecular aggregates during a film-forming process." (1, viewgraphs)

The objective of this Consort 1 experiment was to study the role of convection in evaporative film formation.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (two other experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments including this one) and (b) determine the effect of reentry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the ap-

propriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for this and other film formation and membrane casting experiments. This experiment was allotted five "Type 4" test wells. Discussion of this specific well-type is detailed here.

Each "Type 4" test well provided the investigator with one experimental opportunity. The well-type used four sample wells, one on the top block and three on the bottom block. References (1) and (5) appear to differ on the specific materials which were placed in each well for this experiment. It is suspected that (a) the well in the top block of each well-type contained the membrane forming surface (either glass or a ceramic disc), (b) the first well in the bottom block of each well-type contained a solution of polyperfluoro sulfonic acid, 5% in isopropanol, methanol, and water, (c) the second well in the bottom block of each well-type contained air, and (d) the third well in the bottom block of each well-type contained water.

Prior to flight, the blocks were joined together such that the well in the upper block was aligned with the first well in the bottom block. Thus, prior to and during launch, the solution was in contact with the film-forming surface. Once the rocket had achieved the low-gravity phase, a motor and cam mechanism moved the upper block to the right, aligning the well on the upper block with the well on the lower block which contained air. Although it was not specifically stated in the available publications, the air most likely dried the polymer solution forming the membrane. Just prior to the termination of the low-gravity phase of the rocket, the upper block again moved to the right, now aligning the well on the top block with the well on the bottom block which contained water. The blocks remained in this position throughout the re-entry period.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected and membranes were cast in the "Type 4" cells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Post-flight evaluation of the sample wells was initiated. "Membranes of chitosan [The quote is also referring to another of Pellegrino's Consort experiments (see Pellegrino, Consort 1, Chitosan Membrane Formation)] and perfluorosulfonic acid (Nafion) and composites of these two polymers were cast on 3-mm ceramic discs and examined by scanning electron microscopy. These are currently undergoing comparison with their counterparts cast in the presence of gravity.... There is a substantial difference in the thickness of [Nafion] membranes formed in the two cases." (3, p. 7)

No further information discussing specific experiment objectives or results could be located at this time.

Key Words: Biotechnology, Membranes, Thin Film Membranes, Polymer Membranes, Convection, Porosity, Macromolecular Aggregates, Buoyancy Effects Diminished, Evaporation, Mass Transfer, Medical Applications, Pharmaceutical Applications, Contamination Source, Liquid Leakage

Number of Samples: five
Sample Materials: The top and bottom well allocations as stated in References (1) and (5) and inferred in (3) seem to differ. Presumably-top wells: glass or ceramic discs; bottom wells: (1) polyperfluoro sulfonic acid, 0.5% in isopropanol, methanol and water, (2) air, (3) water.
Container Materials: inert material

Experiment/Material Applications:
"Potential future commercial applications are: medical, pharmaceutical, industrial and scientific industries that have a need for filtration, purification technology, separation of... fluids, etc." (1, viewgraphs)

No specific application of the nafion was detailed.

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight, brief discussion of results)

(4) Letter from J. J. Pellegrino to J. M. Cassanto (ITA). April 12, 1989, describing "Quick Look at CONSORT 1 MDA Results-NIST Membrane Formation." (post-flight; information sent from ITA to C. A. Winter, NASA)

(5) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (Provided by ITA to C. Winter, 1/91)

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Co-Investigator(s): Luttges, M. W. (2), Hayes, J. W. (3), Moos, P. J. (4)
Affiliation(s): (1,3,4) Bioserve Space Technologies, National Aeronautics and Space Administration (NASA) Center for Commercial Development of Space (CCDS) at the University of Colorado, Boulder, Colorado; (2) During Consort 1: Bioserve Space Technologies, NASA CCDS, University of Colorado, Boulder, Colorado, Currently: Deceased

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab)
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Fibrin Clot Formation

"A variety of different biological structures are formed by the directed assembly of large homogeneous protein macromolecules. Many of these processes have been simulated successfully in vitro. Yet, the products of such simulations often lack the specificity and material characteristics of the structures formed within biological systems....

"Presumably, the mismatch between in vivo and in vitro products derives, in part from biophysical differences in the assembly environments. Whereas the in vivo assembly occurs in viscous template-directed fashion, the in vitro assembly occurs in an environment with large scale bulk fluid motions, density driven flows and shear flows. The latter assembly conditions should foster disorder during macromolecule assembly unless template constraints are very stringent." (1, p. 1)

This experiment (together with those of Luttges, Consort 1 and related KC-135 experiments), "...focuses upon using reduced gravity environments to evaluate in vitro macromolecule assembly processes and products. The experiments were designed to evaluate the effects of the reduced gravity assembly conditions on rates of assembly and product characteristics." (1, p. 1) More specifically, this Consort 1 fibrin clot formation experiment was designed to (a) determine the role of convection in blood clot formation and (b) form low-gravity fibrin structures.

During the formation of fibrin in the low-gravity environment, bulk fluid convective mixing, shear forces and material sedimentation should be reduced. Such a reduction of these gravity-induced phenomena should result in "...a slower more homogeneous formation of biomaterial aggregates." (4, viewgraphs)

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two Teflon^{TM} blocks. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto et al., Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted two "Type 3" test wells. Discussion of this specific well-type is detailed here.

Each "Type 3" test well provided the investigator with one experimental opportunity. The well-type used three sample wells, two in the top block and one in the bottom block. Thirty-six

hours prior to the rocket launch, the well in the lower block of each well-type was filled with fibrinogen solution. Twelve hours later, one upper well of each well-type was filled with thrombin and the second upper well of each well-type was filled with 2% glutaraldehyde fixative. The long delay in the filling of the upper and lower wells "...was primarily due to the relatively large number of wells that had to be precisely loaded." (1, p. 3)

Reportedly, the fluids used in the investigation were chosen to match the opportunities/restraints of the Consort 1 rocket and the MDA. "Early" sample loading of the MDA (24-48 hours prior to launch), processing temperature of the samples (approximately 22 ^{o}C), high g loads experienced by the vehicle during takeoff and landing (up to 20 g's), 10^{-5}-10^{-6}g low-gravity duration (approximately 7 minutes), and "late" retrieval of samples (approximately 4 hours post-flight) limited fluid selection and investigative objectives.

Prior to flight, the blocks were joined together such that the wells in the upper block were purposely misaligned with the wells in the lower block. This prevented materials within the wells from coming into contact prior to the initiation of the experiment. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells containing the fibrinogen solution and thrombin. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity phase, the upper block again moved to the right. This movement aligned the well which originally contained only fibrinogen solution (but now contained a mixture of fibrinogen solution and thrombin) with the well containing the liquid glutaraldehyde fixative. These wells remained in contact throughout the re-entry period of the rocket.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 3" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Post-flight analysis of the contents of the wells was performed. Reportedly, "Because of steep concentration gradients across the interface of the precursor and initiator solutions, diffusion was expected to occur at relatively high rates. High rates of diffusion were essential to maximize the amount of material polymerized fibers to remain in the precursor well since fibers would exhibit very high molecular weights and thus would not diffuse away from the assembly location.

"As expected, plugs of fibrin materials... were localized to the well originally containing the precursor solutions. Plugs extended to approximately half of the 16 mm depth suggesting that this was the depth reached by diffusion initiator components. The plugs of material most importantly, were of sufficient size and quality to permit subsequent analysis steps.... Since the plugs have depths that are quite large compared to fiber lengths, an important series of questions can be addressed. These questions relate to the diffusion distances supported by these periods of high-quality low gravity and to any intrinsic effect that diffusion may have on the polymerization process in these sensitive reactions." (3, p. 6) <Note: Which solutions were the precursor solutions and which were the initiator solutions were not specified.>

Gross observations of the opacity of the fibrin indicated that the assembly of the three-dimensional lattice appeared complete.

No further information discussing the experimental results could be located at this time.

Key Words: Biotechnology, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Macromolecular Assembly, Protein Macromolecules, Biological Structures, Fibrin Structures, Blood Clotting, Convection, Polymerization, Mass Transfer, Shear Forces, Sedimentation, Sample Homogeneity, Biological Materials Processing, Structure Based Drug Design, Medical Applications, Contamination Source, Liquid Leakage, Contained Fluids

Number of Samples: two
Sample Materials: Top wells: (1) thrombin, (2) fixative (glutaraldehyde); bottom well: fibrigen solution
Container Materials: Teflon™

Experiment/Material Applications:
"This research will lead to a greater understanding of the effects of gravity on biomaterial processing.... This understanding should lead to novel earth or space-based processes for production of biomaterials." (4, viewgraphs)

More specifically, the experiment had potential commercial applications in the area of blood clotting.

"Fibrin is the major structural protein that forms blood clots." (4, viewgraphs)

References/Applicable Publications:

(1) Moos, P. J., Hayes, J. W., Stodieck, L. S., and Luttges, M. W.: Macromolecular Assemblies in Reduced Gravity Environments. AIAA 28th Aerospace Sciences Meeting, January 8-11, 1990, Reno, Nevada, AIAA 90-0027, 8 pp. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results - A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Electrophoretic Transfer

This Consort 1 electrophoretic transfer experiment was designed to study particle motion in an electromechanical environment.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (three other experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments) the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the

other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted five "Type 2" test wells. Discussion of this specific well-type is detailed here.

Each "Type 2" test well provided the investigator with one experimental opportunity. The well-type used two sample wells, one on the top block and one on the bottom block. Prior to flight, the well in the upper block of each well-type was filled with polyethylene glycol 6.5% in 0.1M water, and the well in the lower block of each well-type was filled with dextran (8.0% in 0.1M phosphate buffer) and water. <Note: It appears that a 4% trypan blue trace dye was also added to the dextran/water mixture (Reference (5)).> The blocks were then joined together such that the well in the upper block was purposely misaligned with the well on the lower block. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity rocket phase, the upper block again moved to the right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Optical density measurements of each well-pair performed after the rocket flight illustrated the amount of dye which had diffused during the low-gravity phase of the rocket.

Very little discussion of results could be located at this time. However, it was reported that "Adding 0.1 molar phosphate ions enhances the transport of charged dye both in the laboratory and

in low gravity. This indicates that an electrokinetic potential across the interface genuinely exists and that the observed electrophoresis of dye ions across the interface is gravity independent, which it should be in the case of these low-molecular weight solutes." (3, p. 7)

No further information discussing specific experiment objectives or results could be located at this time.

Key Words: Biotechnology, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Electrophoresis, Mass Transfer, Particle Motion, Electrochemical Environment, Electrokinetic Potential, Contamination Source, Liquid Leakage, Contained Fluids

Number of Samples: unknown
Sample Material: Top Wells: polyethylene glycol 6.5 % in 0.1 M water. Bottom Wells: dextran, 8.0% in 0.1 M phosphate buffer, water. (It appears that a 4% trypan blue dye was added to the dextran/water mixture (Reference 5).)
Container Materials: inert material

Experiment/Material Applications:
A brief note in Reference (1) indicated that this investigation had commercial applications in the area of bioseparation technology.

No further information discussing the applications of this investigation could be found.

References/Applicable Publications:
(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results- A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter on 1/91)

(5) Letter from P. Todd to J. M. Cassanto (ITA) dated May 9, 1989 which described experimental analysis to date. (provided by ITA to C. A. Winter, 1/91)

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Principal Investigator(s): Todd, P. (1)
Co-Investigator(s): Unknown
Affiliation(s): (1) During Consort 1: National Institute of Standards and Technology, Boulder, Colorado, Currently: University of Colorado Department of Chemical Engineering, Boulder, Colorado

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Phase Rearrangement

This Consort 1 phase rearrangement experiment was designed to study the role of wetting in immiscible fluid transfer. The immiscible fluid system employed (polyethylene glycol and dextran) has specific uses in the area of bioseparation technology.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (three other experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments) the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b)

misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted five "Type 2" test wells. Discussion of this specific well-type is detailed here.

Each "Type 2" test well provided the investigator with one experimental opportunity. The well-type used two sample wells, one on the top block and one on the bottom block. Prior to flight, the well in the upper block of each well-type was filled with 6.3% polyethylene glycol and the well in the lower block of each well-type was filled with 8% dextran in water with a trace dye. The blocks were then joined together such that the well in the upper block was purposely misaligned with the well on the lower block. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity rocket phase, the upper block again moved to the right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Optical density measurements of each well-pair performed after the rocket flight illustrated the amount of dye which had diffused during the low gravity phase of the rocket.

Very little discussion of results could be located at this time. However, it was reported that "...[the] transport [of] dye across an interface between PEG-rich and dextran-rich immiscible aqueous

solutions is the same on earth and under low gravity." (3, p. 7) A short report detailed in Reference (3) appears to couple the results of this experiment with two others by Todd (see also Todd, Consort 1, Capillary Flow; Todd, Consort 1, Turbulent Mixing). "Steep surface-tension gradients were established in two types of transport experiments involving two fully-enclosed liquids with an interface between them. When the two liquids were immiscible aqueous solutions [Phase Rearrangement Experiment], no evidence of capillary flow or reorientation of the phases was obtained during the low-gravity period. When the two liquids were miscible aqueous solutions (one with detergent [Capillary Flow Experiment] and one without [Turbulent Mixing Experiment]) there was also no capillary flow. This last observation could not be made definitely on the ground, where transport was dominated by convection, owing to the similar densities of the two solutions." (3, p. 7).

No further information discussing specific experiment objectives or results could be located at this time.

Key Words: Biotechnology, Phase Partitioning, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Immiscible Fluids, Aqueous Solutions, Capillary Flow, Wetting, Mass Transfer, Separation of Components, Phase Separation, Two-Phase System, Medical Applications, Liquid Leakage, Contamination Source, Contained Fluids

Number of Samples: five
Sample Materials: Top wells: 6.3% polyethylene glycol; bottom wells: 8% dextran in water with a trace dye (the trace dye was most likely trypan blue (4%))
Container Materials: inert material

Experiment/Material Applications:
A brief note in Reference (1) indicated that this investigation had commercial applications in the area of bioseparation technology.

No further information discussing the applications of this investigation could be found.

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter on 1/91)

(5) Letter from P. Todd (NIST) to J. M. Cassanto (ITA) dated May 9, 1989 which described experimental analysis to date. (provided by ITA to C. A. Winter, 1/91)

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Principal Investigator(s): Vera, I. (1)
Co-Investigator(s): None
Affiliation(s): (1) During Consort 1: Decision Analysis Corporation of Virginia, Vienna, Virginia, Currently: Department of Energy, Washington, DC

Experiment Origin: USA and Venezuela
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Cellulose Acetate Membrane Formation

"Thin film membranes cast on earth are subjected to convection forces which are believed to accentuate nonuniformity and variations in both porosity and macromolecular aggregation." (1, viewgraphs) Such anomalies may occur during "Evaporation of [the] solvent and hydration of [the] thin film membranes [which] are convection-dependent processes." (8, viewgraphs)

It was expected that in a low-gravity environment, such buoyancy-driven convection should be reduced and "...membranes with altered characteristics such as higher density and fewer macrovoids" (8, viewgraphs) could be produced. Such production could help to illustrate "...how gravity effects the formation of macromolecular aggregates during a film forming process." (1, viewgraphs)

The objectives of this Consort 1 experiment were to (a) study the role of convection in polymer membrane casting and (b) cast cellulose acetate semipermeable polymer membranes in a low-gravity environment.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (one other experiment, Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments),

the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments including this one) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for this and other film formation and membrane casting experiments.

This experiment was allotted two "Type 4" test wells. Each "Type 4" well provided the investigator with one experimental opportunity. The well-type used four sample wells, one on the top block and three on the bottom block. References (1) and (7) appear to differ on the specific materials placed into each well for the experiment. However, the Principal Investigator verified that (a) the well in the top block of each well-type contained the membrane forming surface (glass), (b) the first well in the bottom block of each well-type contained a polymer solution of cellulose acetate, 2% in acetone and water, (c) the second well in the bottom block of each well-type contained air, and (d) the third well in the bottom block of each well-type contained water.

Prior to flight, the blocks were joined together such that the well in the upper block was aligned with the first well in the bottom block. Thus, prior to and during launch, the polymer solution was in contact with the film-forming surface. Once the rocket had achieved the low-gravity phase, a motor and cam mechanism moved the upper block to the right, aligning the well on the upper block with the well on the lower block which contained air. Although it was not specifically stated in the

available publications, the air most likely dried the polymer solution forming the membrane. Just prior to the termination of the low-gravity phase of the rocket, the upper block again moved to the right, now aligning the well on the top block with the well on the bottom block which contained water. The blocks remained in this position throughout the re-entry period.

"After disassembling of the MDA unit (post-flight), samples were not found in the four glass surfaces originally designed for these membranes. Nevertheless, very small and irregular samples were collected from the surfaces surrounding the wells. Scanning Electron Microscopy (SEM) analysis has been performed on these samples. The results of the analysis show no structural difference between the samples obtained from the Consort I flight and ground control samples cast with a similar MDA unit." (5, p. 1)

Evaluation of the operation of the MDA indicated that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations. "The leak problems of the MDA and the results of this analysis suggest that the samples from Consort I were in fact formed before the rocket was exposed to microgravity." (5, p. 1)

No further information discussing specific experiment objectives or results is available at this time.

Key Words: Biotechnology, Membranes, Thin Film Membranes, Polymer Membranes, Semipermeable Membranes, Porosity, Macrovoids, Evaporation, Hydration, Macromolecular Aggregates, Convection, Casting, Medical Applications, Separation of Components, Filtration, Pharmaceutical Applications, Liquid Leakage, Contamination Source

Number of Samples: two
Sample Materials: Top wells: glass; bottom wells: (1) cellulose acetate, 2% in acetone and water, (2) air, (3) water
Container Materials: inert material

Experiment/Material Applications:
"These type [sic] of membranes have potential applications in a number of advanced technologies including kidney dialysis, gas separations, desalination of sea water, etc." (4)

"Potential future commercial applications are: medical, pharmaceutical, industrial and scientific industries that have a need for filtration, purification technology, separation of...fluids, etc." (1, viewgraphs)

"By casting the membranes in a microgravity environment, the effect of convection upon these polymer thin films can be identified. Furthermore, membranes with a better morphological structure and membranes with fewer imperfections might be produced." (6, p. 65)

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight; no specific experiment results detailed)

(4) Letter from I. Vera to J. Cassanto, January 23, 1988. (preflight; information sent from ITA to C. A. Winter (NASA) on 1/91)

(5) Letter from I. Vera to P. Todd, dated June 8, 1989. Subject: Results from membrane casting experiments on the Consort I sounding rocket. (post-flight, information sent by ITA to C. A. Winter, 1/91)

(6) Verá, I.: The Casting and Mechanism of Formation of Semi-Permeable Polymer Membranes in a Microgravity Environment. Adv. Space Res., Vol. 6, No. 5, pp. 65-68. (related research; discusses future Get Away Special Experiment)

(7) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter, 1/91.)

(8) MDA Experiment No. 16 on Consort 1 and 2 Rocket Flights, Casting of Cellulose Membrane. Information supplied by ITA describing experiment objective and rationale, received 1/91 by C. A. Winter. (preflight)

(9) Input received from Principal Investigator I. Vera, August 1993.

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Principal Investigator(s): Vera, I. (1)
Co-Investigator(s): None
Affiliation(s): (1) During Consort 1: Decision Analysis Corporation of Virginia, Vienna, Virginia, Currently: Department of Energy, Washington, DC

Experiment Origin: USA and Venezuela
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Polysulfone Membrane Formation

"Thin film membranes cast on earth are subjected to convection forces which are believed to accentuate nonuniformity and variations in both porosity and macromolecular aggregation." (1, viewgraphs) Such anomalies may occur during "Evaporation of [the] solvent and hydration of [the] thin film membranes [which] are convection-dependent processes." (8, viewgraphs)

It was expected that in a low-gravity environment, such buoyancy-driven convection should be reduced and "...membranes with altered characteristics such as higher density and fewer macrovoids" (8, viewgraphs) could be produced. Such production could help to illustrate "...how gravity effects the formation of macromolecular aggregates during a film forming process." (1, viewgraphs)

The objectives of this Consort 1 experiment were to (a) study the role of convection in polymer membrane casting and (b) cast polysulfone semipermeable polymer membranes in a low-gravity environment.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11)); Vera, Consort 1 (one other experiment, Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments),

the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments including this one) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for this and other film formation and membrane casting experiments. This experiment was allotted two "Type 4" test wells. Discussion of this specific well-type is detailed here.

Each "Type 4" test well provided the investigator with one experimental opportunity. The well-type used four sample wells, one on the top block and three on the bottom block. References (1) and (7) appear to differ on the specific materials which were placed in each well for the experiment. However, the Principal Investigator verified that (a) the well in the top block of each well-type contained the membrane forming surface (glass), (b) the first well in the bottom block of each well-type contained a polymer solution of polysulfone, 15% in tetrahydrofuran, (c) the second well in the bottom block of each well-type contained air, and (d) the third well in the bottom block of each well-type contained water.

Prior to flight, the blocks were joined together such that the well in the upper block was aligned with the first well in the bottom block. Thus, prior to and during launch, the polymer solution was in contact with the film-forming surface. Once the rocket had achieved the low-gravity phase, a motor and cam mechanism moved the upper block to the right, aligning the well

on the upper block with the well on the lower block which contained air. Although it was not specifically stated in the available publications, the air most likely dried the polymer solution forming the membrane. Just prior to the termination of the low-gravity phase of the rocket, the upper block again moved right, now aligning the well on the top block with the well on the bottom block which contained water. The blocks remained in this position throughout the re-entry period.

Reportedly, "After disassembling of the MDA unit (post-flight), samples were not found in the four glass surfaces originally designed for these membranes. Nevertheless, very small and irregular samples were collected from the surfaces surrounding the wells. Scanning Electron Microscopy (SEM) analysis has been performed on these samples. The results of the analysis show no structural difference between the samples obtained from the Consort I flight and ground control samples cast with a similar MDA unit." (5, p. 1)

Evaluation of the operation of the MDA indicated that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations. "The leak problems of the MDA and the results of this analysis suggest that the samples from Consort I were in fact formed before the rocket was exposed to microgravity." (5, p. 1)

No further information discussing specific experiment objectives or results is available at this time.

Key Words: Biotechnology, Membranes, Thin Film Membranes, Polymer Membranes, Semipermeable Membranes, Porosity, Macrovoids, Evaporation, Hydration, Macromolecular Aggregates, Convection, Casting, Medical Applications, Separation of Components, Filtration, Pharmaceutical Applications, Liquid Leakage, Contamination Source

Number of Samples: two
Sample Materials: Top wells: glass; bottom wells: (1) polysulfone, 15% in tetrahydrofuran, (2) air, (3) water
Container Materials: inert material

Experiment/Material Applications:
"These type [sic] of membranes have potential applications in a number of advanced technologies including kidney dialysis, gas separations, desalination of sea water, etc." (4)

"Potential future commercial applications are: medical, pharmaceutical, industrial and scientific industries that have a need for filtration, purification technology, separation of... fluids, etc." (1, viewgraphs)

"By casting the membranes in a microgravity environment, the effect of convection upon these polymer thin films can be identified. Furthermore, membranes with a better morphological structure and membranes with fewer imperfections might be produced." (6, p. 65)

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Letter from I. Vera to J. Cassanto, January 23, 1988. (preflight; information sent from ITA to C. A. Winter (NASA) on 1/91)

(5) Letter from I. Vera to P. Todd, dated June 8, 1989. Subject: Results from membrane casting experiments on the Consort I sounding rocket. (post-flight; information sent by ITA to C. A. Winter, 1/91)

(6) Vera, I.: The Casting and Mechanism of Formation of Semi-Permeable Polymer Membranes in a Microgravity Environment. Adv. Space Res., Vol. 6, No. 5, pp. 65-68. (related research; discusses future Getaway Special Experiment)

(7) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter, 1/91.)

(8) MDA Experiment No. 16 on Consort 1 and 2 Rocket Flights: Polysulfone Membrane Casting. Information supplied by ITA describing experiment objective and rationale, received 1/91 by C. A. Winter. (preflight)

(9) Input received from Principal Investigator I. Vera, August 1993.

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CHAPTER 2

CAPILLARITY

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Experiment Origin: USA
Mission: Skylab SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: December 1973, January 1974 (months during mission experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Skylab Student Project, High School Student Experiment, Skylab Manned Environment
Processing Facility: Two capillary tube modules and one capillary wick module
Builder of Processing Facility: NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Capillary Study (ED72)

"On Earth, capillary effects are observed when fluid adhesion to a solid surface produces a force that is sufficient to support the weight of the column of fluid. If the fluid wets the surface of a small capillary tube, it is drawn up [rises] until the weight of the column equals the adhesion force." (4, p. 84) Because gravity is inversely proportional to this rise height, it has been theorized that the capillary rise might tend to infinity under low-gravity conditions.

The objective of this Skylab SL-4 experiment was to determine if capillary and wicking actions in space differ from those observed on Earth.

The Skylab SL-4 experiment apparatus consisted of a two capillary tube modules and one capillary wick module. Each capillary tube module contained (1) three capillary tubes (of graduated sizes) and (2) a fluid reservoir (composed of a force-free bladder). (The two capillary tube modules were identical except that one reservoir contained Krytox oil and the other contained water.) The capillary wick module consisted of (1) three capillary tubes (of twill and mesh screens) and (2) a reservoir (which contained water with a wetting agent). The wetting agent was added to produce a surface tension-to-density ratio similar to hydrogen. <Note: Further descriptions of these modules could not be lo-

cated.>

"During the experiment, the mouths of the capillary instruments were to be kept in contact with the reservoir fluid, but the capillary action was to be prohibited by a specific valve until the experiment was activated by a Skylab crewman." (4, p. 85) The experiment was photographed with a remotely operated 16 mm camera equipped with an 18 mm lens. (All fluids used for this experiment contained a coloring agent for photographic contrast.)

The valve in the capillary wick module was opened first. Little or no wicking action was observed in the module for more than 2.5 hours. At this time, it was necessary to move the module. The agitation from this movement apparently initiated some minor wicking action.

Several days later, the capillary tube modules were activated. However, no capillary action was observed in these modules.

"In an effort to find the cause for such a lack of action, the crew discovered evidence of fluid leakage from the reservoirs...." (4, p. 85) Apparently, this leakage occurred prior to the beginning of the experiment. "The loss [of fluid] could have been caused by vibrations of the Saturn [rocket] during launch or by the excessive temperatures and reduced pressure [resulting in a boiling of the fluid] during the early days of the... Skylab mission." (4, p. 85)

<Note: Reference (5), as listed below, was not available to aid in the preparation of this summary.>

Key Words: Capillarity, Capillary Flow, Capillary Rise, Capillary Tubes, Capillary Forces, Hydrodynamics, Wicking, Free Surface, Surface Tension, Wetting of Container, Wetting Agent, Liquid Reservoir, Liquid Transfer, Liquid/Gas Interface, Solid/Liquid Interface, Liquid Leakage, Thermal Environment More Extreme Than Predicted, Rocket Motion, Liquid Vibration, Boiling, Gas Pressure, Rocket Vibration, Processing Difficulties

Number of Samples: nine capillary tubes
Sample Materials: Fluid reservoirs of the capillary tube modules: (1) water, (2) Krytox oil; fluid reservoir of the capillary wick module: "...water with a wetting agent to produce a surface tension-to density ratio simulating liquid hydrogen." (3, pp. 6-47 and 6-50)

Container Materials: Capillary tube modules: unknown; capillary wick modules: twill and mesh screens.

Experiment/Material Applications:
Research applications of this experiment include (1) substantiating theoretical capillary rise fluid flow models with experimental data, and (2) understanding and predicting fluid behavior under low-gravity conditions (e.g., rocket propellant motion).

The water/wetting agent combination was used to simulate liquid hydrogen, which is a widely used rocket fuel.

References/Applicable Publications:
(1) "Capillary Study (ED72)." In MSFC Skylab Mission Report-Saturn Workshop, Skylab Program Office, George C. Marshall Space Flight Center, Alabama, October 1974, NASA TM X-64814, pp. 12-82 - 12-83. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) "Experiment ED72 - Capillary Study." In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 6-47 - 6-53. (post-flight)

(4) Skylab, Classroom in Space. Edited by: L. B. Summerlin, NASA SP-410, pp. 84-85. (post-flight)

(5) Johnston, R. G.: ED-72 Final Report, Marshall Space Flight Center.

(6) Input received from Principal Investigator R. G. Johnston, June 1989.

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Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, ASTP Manned Environment
Processing Facility: Foaming solutions were initiated into test tubes via syringes
Builder of Processing Facility: Unknown

Experiment:
The Formaldehyde Clock Reaction/Chemical Foaming

This experiment was one of two chemical foam science demonstrations designed by Grodzka et al. performed during the ASTP mission. (See Grodzka, ASTP, "Equilibrium Shift Reaction/Chemical Foaming" to review the other experiment.) The specific objective of the formaldehyde clock reaction experiment was to determine the low-gravity reaction times of certain chemical mixtures when these mixtures were altered from bulk liquid to stable foam. (A detailed discussion of the stability of foams is detailed under Grodzka, ASTP, "Equilibrium Shift Reaction/Chemical Foaming.")

The formaldehyde clock reaction was selected for study because the reaction (1) appeared to involve surface adsorption and (2) allowed the collection of quantitative data using a simple procedure.

The reaction occurs when the following aqueous solutions are mixed together: (1) formaldehyde, (2) phenolphthalein indicator, (3) sodium sulfite, and (4) sodium bisulfate. The mixture remains colorless for a period of time until the reaction is completed. The color of the solution at the time of completion is vivid red. The reaction times are a function of the reacting species concentration. (See Reference (2) for a complete listing of solution compositions, procedures and reactions.)

During the ASTP experiment, four tubes (each containing all four of the reacting solutions) were shaken vigorously for approximately 5 seconds by a crew member. Approximately 20-30 seconds later, the colorless liquid foamed and turned bright red.

"The color change began as a red ring and rapidly spread through the liquid." (3, 31-4) The process was recorded on motion picture film.

It was reported that "Because of various circumstances... data obtained on the time intervals between reaction initiation and reaction end were not sufficiently precise to allow an unequivocal decision on whether or not the reaction proceeded more rapidly in space than on the ground. The motion pictures, however, showed that true foams were not obtained in the low-g tests. Stable gas/liquid dispersions, however, were. Also, the reaction preferentially started at the liquid meniscus... and not in the gas/liquid dispersion as had been expected." (2, p. 2)

This unexpected surface effect spurred further ground-based experiments. These experiments led to the following conclusions: (1) "Adsorption of dissociated and undissociated formaldehyde species at solid/liquid and gas/liquid surfaces plays an important role in the reaction...." (2, p. 2) (2) Surprisingly, "Reaction rates are apparently affected by the volume/surface-area ratios of the reacting liquids and the nature and extent of all surface interfaces, indicating the operation of long-ranged forces." (2, p. 2)

Key Words: Capillarity, Foams, Foam Stability, Aqueous Solutions, Reactant Solutions, Liquid Mixing, Bubbles, Bubble Dispersion, Liquid/Gas Dispersion, Liquid/Gas Interface, Interface Physics, Stability of Dispersions, Surface Tension, Container Shape, Crucible Effects, Contained Fluids, Solid/Liquid Interface, Liquid Expulsion Through a Small Orifice, Blood Clotting

Number of Samples: four
Materials: A solution comprised of: (1) sodium sulfite, (2) sodium metasulfite, formaldehyde, and phenolphthalein indicator (see Reference (1) for details)
Container Materials: Lexan™ test tubes

Experiment/Material Applications:
This experiment is related to fundamental research in the area of biophysical processes. The formaldehyde clock reaction is relevant to phenomena such as blood clotting and caratact formation. (See Reference (1) for more details.)

Other reasons why the formaldehyde reaction was chosen for the experiment are detailed in the above experiment summary.

See also Grodzka, ASTP, "Equilibrium Shift Reaction/Chemical Foaming."

References/Applicable Publications:

(1) Grodzka, P.: Three Model Space Experiments on Chemical Reactions. Proc. of the 1976 NASA Colloquium on Bioprocessing in Space, Houston, Texas, March 10-12, 1976, NASA TM X-58191, pp. 67-76. (post-flight)

(2) Grodzka, P. and Facemire, B.: Chemical Reactions in Low-G. American Institute of Aeronautics and Astronautics 16th Aerospace Sciences Meeting, Huntsville, Alabama, January 16-18, 1978, 5 pp. (post-flight)

(3) Snyder, R. S., Clifton, K. S., Facemire, B., Whitaker, A. F., Grodzka, P. G., and Bourgeois, S.: Science Demonstrations. In Apollo-Soyuz Test Project--Preliminary Science Report, NASA TM X-58173, February 1976, pp. 31-1 - 31-9. (post-flight) <Note: This document is not the same as the special publication entitled: Apollo-Soyuz Test Project.>

(4) Grodzka, P. G. and Bourgeois, S. V.: The Apollo-Soyuz Science Demonstration Experiments, Final Report. Lockheed Missiles and Space Co. Report, LMSC-HREC-TR-D497499, October 1977, work performed under NASA/MSFC Contract NAS8-32222. (post-flight)

(5) Grodzka, P. G. and Facemire, B.: Surface and Internal Structure Effects in the Formaldehyde Clock Reaction. Submitted to Science January 19, 1976. <Note: The current status of this document is unclear at this time.>

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Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, ASTP Manned Environment
Processing Facility: Foaming solutions were initiated into test tubes via syringes
Builder of Processing Facility: Unknown

Experiment:
Equilibrium Shift Reaction/Chemical Foaming

"A foam consists of bubbles of gas encased by walls of thin liquid films. Although some foams can be made rigid or flexible and durable, the characteristic foam is not stable on Earth. Foams collapse primarily as a result of the gravity-induced drainage of liquid in the bubble walls. As the liquid drains from the bubble, the film walls become increasingly thin until, at a thickness of approximately 10 nanometers (100 angstroms), they are ruptured by random molecular motion. Thus, the foam gradually dissipates as more of the liquid drains away to bulk form and leaves less to support the bubble walls. In the zero-g environment of space, drainage is substantially reduced; therefore, a longer lasting foam is provided. Drainage, however, is only one of the mechanisms contributing to the dissipation of foams. Evaporation and liquid spreading also act to dissipate bubble walls. Thus, the full extent of foam stability is still uncertain.

"Forming a foam in space yields a very large surface area-to-volume ratio of the liquid that should be retained for a relatively long period. This large, stable surface area should enable surface-sensitive chemical reactions to occur differently than they would on Earth. The sensitivity of some chemical reactions to the size of the interface area has long been known.... However, the precise change taking place at the interface has not been precisely determined, and definitive theories on the characteristics of a stable foam in space have not been generated. Nevertheless, it was anticipated that a chemical reaction dependent on the surface area of the air-to-liquid interface... would

proceed differently in a foam in a zero-g environment than would normally occur on Earth." (3, pp. 31-2 - 31-3)

This experiment was one of two chemical foam science demonstrations designed by Grodzka et al. performed during the ASTP mission. (See Grodzka, ASTP, "The Formaldehyde Clock Reaction/Chemical Foaming" to review the other experiment.) The specific objective of the Equilibrium Shift Reaction Experiment was to visually demonstrate the stability of foams under low-gravity conditions.

Prior to the initiation of the experiment, it appears that two Lexan™ test tubes were each filled with a gold-colored solution comprised of (1) 10.7 mg thymol blue, (2) 10 ml ethyl alcohol, (3) 0.2 cc concentrated hydrochloric acid, and (4) 500 ml distilled water. (Thymol blue of pH 2.8 will turn from brown amber to pink when foamed by shaking.)

During the ASTP experiment, a crew member shook the tubes and the resulting pink foams were recorded on motion picture film. The contrast of the brightly colored pink foam against the gold bulk solution was clearly visible to the crewman as the foam dissipated.

It was reported that (a) the foams created under low-gravity conditions were very stable while those foams created on Earth collapsed within a few seconds, and (b) because the "...16 millimeter motion picture was out of focus, detailed knowledge of the foam dissipation [time] in the pink foam has not been determined." (3, p. 31-4)

No other results from this experiment were reported.

Key Words: Capillarity, Foams, Foam Stability, Aqueous Solutions, Reactant Solutions, Liquid Mixing, Bubbles, Bubble Dispersion, Surface Tension, Liquid Spreading, Thin Films, Fluid Drainage, Interface Physics, Evaporation, Contained Fluids, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Liquid Expulsion Through a Saml1 Orifice, Photographic Difficulties

Number of Samples: two
Sample Materials: A solution comprised of: (1) thymol blue, (2) water, (3) ethyl alcohol, and (4) hydrochloric acid
Container Materials: Lexan™ test tubes

Experiment/Material Applications:
"Foams are currently used in such diverse applications as the separation of proteins by the fractionation of foams and the origination of rigid structures such as foamed polystyrene and foam rubber. It has been suggested... that such unique products as reinforced rigid foams could be formed in space without the influence of gravity. In such foams, metal fibers would be alined [sic] along the surface of a stable foam strictly by adhesion. When cured, the foam could have a light-weight durability possibly unknown with earthbound processes. It has also been suggested that space-originated foams could act as the basis of porous metal electrodes. Such new products and synthesis procedures are potential applications of foam technology in space." (3, p. 31-3)

References/Applicable Publications:
(1) Grodzka, P.: Three Model Space Experiments on Chemical Reactions. Proc. of the 1976 NASA Colloquium on Bioprocessing in Space, Houston, Texas, March 10-12, 1976, NASA TM X-58191, pp. 67-76. (post-flight)

(2) Grodzka, P. and Facemire, B.: Chemical Reactions in Low-G. American Institute of Aeronautics and Astronautics 16th Aerospace Sciences Meeting, Huntsville, Alabama, January 16-18, 1978, 5 pp. (post-flight)

(3) Snyder, R. S., Clifton, K. S., Facemire, B., Whitaker, A. F., Grodzka, P. G., and Bourgeois, S.: Science Demonstrations. In Apollo-Soyuz Test Project--Preliminary Science Report, NASA TM X-58173, February 1976, pp. 31-1 - 31-9. (post-flight) <Note: This paper is not the same as the special publication entitled: Apollo-Soyuz Test Project.>

(4) Grodzka, P. G. and Bourgeois, S. V.: The Apollo Soyuz Science Demonstration Experiments; Final Report. Lockheed Missiles and Space Company, Inc., LMSC-HREC-TR-D497499, October 1977, work performed under NASA/MSFC Contract NAS8-32222.

(5) Naumann, R. J. and Mason, E. D.: Chemical Foams. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 72. (post-flight)

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Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, ASTP Manned Environment
Processing Facility: Wicks attached to tetrafluoroethylene (Teflon™) base plate
Builder of Processing Facility: Materials and Processing Laboratory, NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Capillary Wicking

In the absence of gravity, fluids can be maneuvered and maintained by passive devices that do not require spacecraft power. "Capillary wicking principles have been used in spacecraft for containment of fuel/cryogen fluids, separation of liquid-vapor phases, and expulsion of propellants. Little is known, however, about the efficiency of transfer and the wicking rate of stainless steel wicks used during space missions...." (1, p. 31-6.) Reduced gravity experiments performed in drop tower facilities or on KC-135 parabolic aircraft flights have not been of long enough duration to determine basic wicking design criteria.

Theoretical determinations of liquid movement through the wick are limited to ideal wicks possessing evenly spaced, parallel fibers. These calculations cannot be directly applied to realistic cloth or metal-fabric wicks which have variable properties.

This ASTP Science Demonstration Experiment was designed to investigate capillary wicking under low-gravity conditions. The specific objective of the investigation was to determine the wicking rates and efficiency of fluid transfer of wicks used for fluid management in spacecraft.

Four identical wicking assemblies were used during the experiment. Each assembly contained three stainless steel wicks and one nylon wick. The stainless steel wicks differed in their weave and mesh size: (1) 325 by 2300 Dutch twill weave, (2) 200 by 200 plain weave, and (3) 200 by 600 Plain Dutch weave; the nylon wick was included for comparison. The wicking fluid of one or more of the assemblies was a water-soap solution and the wicking fluid of the remaining assemblies was silicone oil. Blue dye

was added to both fluids for better visibility.

Reportedly, "The wicks were attached to an assembly that was affixed to a tetrafluoroethylene [Teflon™] base plate. A small weld was cut from the center of the tetrafluoroethylene plate, and the fluids were inserted into this well. A rough aluminum surface covering the bottom of the well ensured better retention of the fluid." (1, p. 31-6)

Reportedly, "The wicking of both oil and water proceeded much faster during the ASTP mission than had been anticipated on the basis of ground tests and KC-135... experiments." (1, p. 31-7) "The liquid was observed to rise along the corner formed by the Teflon support back and the mesh. Since Teflon is not normally wetted by the fluids used, this behavior was unanticipated and serves to illustrate how unexpected fluid behavior can occur in weightlessness." (2, p. 74)

No other discussions or results from this experiment could be located.

Key Words: Capillarity, Capillary Flow, Capillary Forces, Hydrodynamics, Aqueous Solutions, Wicking, Free Surface, Surface Tension, Contact Angle, Wetting, Liquid Transfer, Mass Transfer, Liquid Reservoir, Propellant Tanks, Fluid Management, Liquid/Gas Interface, Solid/Liquid Interface, Fibers

Number of Samples: sixteen
Sample Materials: Wicks: (1) stainless steel, (2) cloth; wicking Solutions: (1) silicone oil, (2) low surface tension water-soap solution
Container Materials: tetrafluoroethylene (Teflon™) base plate

Experiment/Material Applications:
Wicking principles are important in several research areas including heat pipe technology, material absorption, liquid transfer, and liquid positioning.

References/Applicable Publications:

(1) Snyder, R. S., Clifton, K. S., Facemire, B., Whitaker, A. F., Grodzka, P. G., and Bourgeois, S.: Science Demonstrations. In Apollo-Soyuz Test Project, Preliminary Science Report, NASA TM X-58173, pp. 31-1 - 31-9, 1976. (post-flight)

(2) Naumann, R. J. and Mason, E. D.: Capillary Wicking. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 74. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 2
Launch Date/Expt Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06 (TEM 06 E)
Builder of Processing Facility: Unknown, possibly, ERNO, Bremen, Germany

Experiment:
Wetting Kinetics/Capillarity

Capillary forces play a major role in the motion of liquids through very small capillaries and pores. Understanding and predicting the role of these forces is difficult because (1) direct observation of the behavior of liquids in such small volumes is usually not possible and (2) mathematical descriptions of the motion are limited because non-cylindrical capillaries of varying geometries are often involved. On Earth, small-pore capillary movement cannot be modeled in larger, observable scale (wide-pore) systems because gravity is the dominating force and capillarity can be ignored. Under low-gravity conditions, capillarity can be studied using a wide-pore system.

This TEXUS 2 experiment was the first in a series of investigations designed by Sell et al. to study capillary forces under low-gravity conditions. The specific objective of the experiment was to observe the rise of water in capillary tubes in a reduced gravity environment. It was expected that this study of capillarity would also lead to a better understanding of wetting kinetics.

The experiment was performed in the TEM 06 Liquid Module. The module contained an experimental apparatus which was equipped with (1) a liquid intake system, (2) a liquid container, (3) liquid rise aids, (4) rise tubes, (5) a rise tube holder, (6) a viewing port, (7) liquid measuring tubes, (8) an illumination system, and (9) a pneumatic system which lowered and raised the measuring tubes. (See Reference (1) for a complete description of the processing facility.)

During the low-gravity phase of the rocket, the container was partially filled (with sloshing minimized) with the measuring liquid (water). (The fluid level of the measuring liquid was

such that the rise tubes contacted the surface of the water.) The measuring tubes were then lowered on top of the rise tubes. A total of five measuring tubes of three geometries were employed (a) one cylindrical tube, (b) one conically constricted tube, and (c) three sinusoidally widening and narrowing tubes. The influx of the measuring liquid into the measuring tubes was filmed.

Post-flight examination of the film revealed that no measuring liquid could be observed entering the tubes. Evaluation of the flight apparatus and flight data indicated that the pneumatic system functioned properly and the measuring fluid was not spilled through sloshing. However, it was determined that the rise tubes had poor wetting characteristics and "...thus the water was unable to penetrate with the aid of capillarity forces." (1, p. 129) ESCA examinations of the rise tubes indicated the presence of a hydrocarbon film coating the rise tube and measuring tube surfaces. "At the present it must be concluded that contamination of the rise and measuring tubes after they had been cleaned had such a negative influence on the wetting characteristics that the experiment could not take place according to plan." (1, p. 130) <Note: "ESCA" was not defined.>

Key Words: Capillarity, Capillary Flow, Capillary Rise, Capillary Forces, Capillary Tubes, Hydrodynamics, Liquid Transfer, Liquid Reservoir, Sloshing, Wetting, Wetting Kinetics, Non-Wetting of Container, Free Surface, Surface Tension, Meniscus Shape, Wide-Pore Systems, Pores, Capillaries, Liquid/Gas Interface, Solid/Liquid Interface, Container Shape, Aspect Ratio, Partially Filled Containers, Coated Surfaces, Thin Films, Contamination Source, Processing Difficulties

Number of Samples: five rise tube/measuring tube combinations
Sample Materials: Measuring fluid: water
Container Materials: Measuring tube material: unknown
Rise tube materials: unknown

Experiment/Material Applications:
"Such observations would be expected to lead to a better understanding of the complex inter-relationships between procedures of wetting kinetics." (1, p. 122)

References/Applicable Publications:

(1) Sell, P. J. and Renzow, D.: Capillarity. Shuttle/Spacelab Utilization Final Report, Project TEXUS, 1978, pp. 121-131. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3
Launch Date/Expt. Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-2
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Wetting Kinetics

This TEXUS 3 experiment was the second in a series of investigations designed by Sell et al. to study capillary forces under low-gravity conditions (see Sell, TEXUS 2). The specific objective of the experiment was to evaluate the wetting kinetics of tubes with different geometries.

A description of the TEXUS 3 experimental setup and in-flight experiment procedure was not presented.

Reportedly, due to a rocket despin failure, TEXUS 3 did not achieve the desired low gravity level. The experiment was reflown on TEXUS 3b (see Sell, TEXUS 3b).

Documentation of the results of this TEXUS 3 experiment does not appear to be available.

Key Words: Capillarity, Capillary Flow, Capillary Forces, Capillary Tubes, Liquid Transfer, Hydrodynamics, Wetting, Wetting Kinetics, Meniscus Shape, Liquid/Gas Interface, Container Shape, Acceleration Effects, Rocket Despin Failure

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
See Sell, TEXUS 2, experiment summary

References/Applicable Publications:
(1) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Center (JSUP). (identifies rocket failure)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3b
Launch Date/Expt Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-2: Fluid Module
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Wetting Kinetics/Capillarity

This TEXUS 3b experiment was the third in a series of investigations designed by Sell et al. to study capillary forces under low-gravity conditions (see Sell, TEXUS 2, TEXUS 3). The specific objective of the experiment was to observe the rise of water in capillary tubes in a reduced gravity environment.

The experiment was carried out in the TEM 06 Fluid Module. "The apparatus consisted of a reservoir equipped with five stand pipes. Once microgravity conditions were established, the reservoir was filled with desalinated water from a supply tank. The stand pipes served to stabilize the free surface of the liquid and to form flat menisci into which capillary glass tubes with cylindrical, sinusoidal or conical shapes were lowered. The rise of the liquid in the glass tubes was filmed with a camera at 200 frames/s. Tracer particles in the liquid allowed the visualization of the velocity field both in the bulk and close to the rising menisci." (7, p. 20) The test chamber was filled with humid air (RH = 70-75%) prior to rocket launch in order to guarantee wetting of the tube walls by liquid.

Research related to the TEXUS experiment was also performed on Earth in a simulated weightlessness liquid/liquid system. The system employed two immiscible liquids of the same density, in a Plateau setup. Wetting kinetic experiments with model tubes of different geometries were performed to (1) study the flow behavior at and near the interface and (2) prepare for the TEXUS experiment scenario. Reportedly, "The evaluation of experiments with liquid-liquid systems shows that in connection with transport processes interfaces are very stable. Furthermore, menisci in cylindrical and conical tubes... are of almost hemisperical shape like those in capillary hydrostatics. Merely in sinusoidally shaped tubes strongly deformed menisci occur at

the bottlenecks." (5, p. 583)

"Experiments performed within the framework of the TEXUS programme with tubes of the same geometry demonstrate that in case of transport processes the interfaces of liquid-liquid systems are less stable, already in round tubes there exist strongly deformed menisci. Due to these deformed interfaces, the capillary force, which is the driving force for the transport process, is reduced. Hence the speeds of rise of the menisci are slower. This means that at the bottlenecks of the sinusoidal tubes the deformations of the menisci are not as distinct as in liquid-liquid systems.... Motion pictures indicate clearly that the liquid flows toward the meniscus in a fountain-like manner, and thus gives rise to stagnation pressure at the interface." (5, p. 583) It was noted that wetting aids mounted in the stand pipes unexpectely increased the convective forces.

"For both systems computer models have been developed which allow... [prediction of] the liquid rise and the wetting kinetics in tubes of different geometry. By inclusion of the stagnation pressure, which is caused by the 'fountain-flow', into the flow model for the determination of the meniscus shape, it can be shown that the deformation of the interface occurs already in the vicinity of the tube wall. Therefore an observer will notice a dynamic contact angle which, however, may be attributed to hydrodynamic forces at the interface." (5, p. 583)

Key Words: Capillarity, Capillary Flow, Capillary Rise, Capillary Forces, Capillary Tubes, Hydrodynamics, Liquid Transfer, Liquid Reservoir, Immiscible Fluids, Wetting, Wetting Kinetics, Wetting of Container, Contact Angle, Meniscus Shape, Meniscus Stability, Free Surface, Surface Tension, Liquid/Gas Interface, Interface Physics, Fountain Effect, Solid/Liquid Interface, Wall Effects, Convection, Container Shape, Tracer Particles, Plateau Setup, Liquid/Liquid Interface

Number of Samples: five stand pipes/capillary tubes combinations
Sample Materials: Measuring fluid: desalinated water
Container Materials: Rise tube material: unknown
Measuring tube material: unknown

Experiment/Material Applications:
See Sell, TEXUS 2.

References/Applicable Publications:
(1) Sell, P. J., Maisch, E., and Siekmann, J.: Experimental Study of Fluid Transport in Capillary Systems. 35th International Astronautical Federation International Astronautical Congress, Lausanne, Switzerland, October 7-13, 1984, IAF Paper #84-143, 8 pp. (post-flight; general discussion of all TEXUS flight experiments)

(2) Sell, P. J., Maisch, E., and Siekmann, J.: Hydrodynamic Forces Resulting from Liquid Motion in Capillary Tubes. In Advanced Space Research, Vol. 4, No. 5, 1984, pp. 49-52. (post-flight; mainly modeling/analysis, TEXUS 3b results briefly discussed)

(3) Maisch, E., Sell, P. J., and Siekmann, J.: über den Anstieg von Flüssigkeiten in vertikalen makroskopischen Modellrohren unter Weltraumbedingungen (On the Rise of Liquid Columns into Vertical Tubes Subjected to Zero Gravity) In Ingenieur-Archiv, 56 (1986), pp. 281-294. (post-flight; in German)

(4) Leiner, W., Schindler, B., and Sell, P. J.: Report on the Influence of Boundary Surface Forces at the Boundaries of Liquid Phases Regarding Processes of Liquid Transport Under Zero Gravity Conditions - Literature Survey. ESA Technical Translation of BMFT-FB-W-77-19, June 1983, ESA-TT-576. (related discussions)

(5) Sell, P. J., Maisch, E., and Siekmann, J.: Fluid Transport in Capillary Systems under Microgravity. In Acta Astronautica, Vol. 11, No. 9, 1984, pp. 577-583. (post-flight)

(6) Liquid Motion in Capillary Tubes. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 20-21. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 5
Launch Date/Expt Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-2: Liquid Module
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Wetting Kinetics/Capillarity

<Note: Very little information was available which described this wetting kinetics/capillary experiment. The information presented below was obtained primarily from Reference (2).>

This TEXUS 5 experiment was the fourth in a series of investigations designed by Sell et al. to study capillary forces under low-gravity conditions (see Sell, TEXUS 2, TEXUS 3, TEXUS 3b). The specific objective of the experiment was to observe the rise of water in capillary tubes in a reduced gravity environment.

The TEXUS TEM 06-2 experiment module was employed for the investigation. During the low-gravity phase of the mission, five glass tubes (conically, cylindrically, or sinusoidally shaped) were lowered into a cuvette. (A planar water/air interface was established in the cuvette by using wetting and non-wetting coatings.) <Note: additional specifics describing these coatings were not presented.> As the end of each tube contacted the water/air interface, water rose up the tube and menisci of different shapes formed. The velocity of the water rise varied depending on the diameter and shape of the tubes.

It was reported that "The behavior of the rising liquid was in good agreement with computer simulations." (3, p. 22)

No other results from the TEXUS 5 experiment could be located. See Reference (2) for discussions of computer model.

Key Words: Capillarity, Capillary Flow, Capillary Rise, Capillary Forces, Capillary Tubes, Hydrodynamics, Liquid Transfer, Wetting, Wetting Kinetics, Wetting of Container, Non-Wetting of Container, Free Surface, Surface Tension, Meniscus Shape, Liquid/Gas Interface, Solid/Liquid Interface, Interface Physics, Container Shape, Coated Surfaces

Number of Samples: five
Sample Materials: desalinated water
Container Materials: glass

Experiment/Material Applications:
See Sell, TEXUS 2.

References/Applicable Publications:
(1) Sell, P. J., Maisch, E., and Siekmann, J.: Experimental Study of Fluid Transport in Capillary Systems. 35th International Astronautical Federation, International Astronautical Congress, Lausanne, Switzerland, October 7-13, 1984, IAF Paper #84-143, 8 pp. (post-flight; general discussion)

(2) Sell, P. J., Maisch, E., and Siekmann, J.: Hydrodynamic Forces Resulting from Liquid Motion in Capillary Tubes. Adv. Space Res., Vol. 4, No. 5, 1984, pp. 49-52. (modeling/analysis rather than space results; post-flight)

(3) Liquid Motion in Capillary Tubes. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 22-23. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 10
Launch Date/Expt Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-2
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Bubbles and Foams

<Note: Very little information was available which described this bubbles and foams experiment. The information presented below was obtained primarily from Reference (2).>

This TEXUS 10 experiment was designed to investigate the formation of foams under low-gravity conditions. The specific objective of the experiment was to study (1) the growth and coalescence of gas bubbles within an aqueous solution and (2) the eventual formation of these bubbles into a polyhedral foam.

Bubbles were formed in each of four experiment cells by injecting gas (through a gas jet nozzle) into aqueous solutions of sodium dodecylsulfate (SES). The bubbles were forced into contact with each other by reducing the amount of liquid in the cells. This process resulted in the formation of a polyhedral foam. The experiment was filmed with a cine camera.

Post-flight, it was reported that "As the bubbles detached from the gas input nozzle relatively easily, it was possible to produce a spherical foam under reduced gravity. It was also possible to change a polyhedral foam to a spherical foam. The latter observation gave information on the role of the Plateau-border, which caused the liquid to be sucked from the reservoir into the foam and lamellae as external forces, due to gravity or artificial low pressure, were reduced." (2, p. 24) <Note: No mention was made of how the foam went from polyhedral to spherical. Further, the exact meaning of the last sentence of the above quote was not clear to the editors.>

Reportedly, corresponding experiments were performed on Earth, which allowed (1) the study of foam stability and (2) the determination of dynamic surface properties. Further details of these ground-based experiments were not presented.

Key Words: Capillarity, Foams, Foam Stability, Gas Injection, Bubbles, Bubble Formation, Bubble Dispersion, Bubble Coalescence, Aqueous Solutions, Surface Tension, Liquid/Gas Interface, Solid/Liquid Interface, Contained Fluids, Liquid Reservoir

Number of Samples: four experiment cells
Sample Materials: four different aqueous solutions of sodium dodecylsulfate; foaming gas: unknown
Container Materials: unknown

Experiment/Material Applications:
The specific reason why sodium dodecylsulfate was used during this experiment was not discussed in the available English literature.

References/Applicable Publications:
(1) Gebhardt, K. F., Renzow, D., and Sell, P. J.: Blasenerzeugung, Blasenkoaleszenz und Schaumstabilität unter Mikrogravitations- Bedingungen (Generation and Coalescence of Bubbles and Stability of Foams under Microgravity Conditions). In BMFT-FB-W 86-020, Final Report, December 1986. (post-flight; in German, with summary translated into English)

(2) Bubbles and Foams. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 24. (post-flight, brief description of experiment)

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Experiment Origin: Spain
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Fluid Physics Module (FPM)
Builder of Processing Facility: FIAT C.R., Italy

Experiment:
Floating Zone Stability in Zero Gravity 1ES331

This Spacelab 1 experiment was the first in a series of investigations designed by Da Riva and/or Martínez et al. to study the formation and stability of a liquid bridge under low-gravity conditions. The specific objectives of the investigation were to (1) form a long cylindrical bridge between two coaxial disks and (2) investigate the surface deformation and liquid flow caused by mechanical stimuli (e.g., rotation, vibration, stretching).

During the experiment, liquid bridges were to be established between two coaxial stainless steel disks configured within the Spacelab Fluid Physics Module (FPM). Several bridges were to be created during the course of the mission with silicone oils of 5 x 10^{-6} m^2/s and 100 m^2/s viscosities. The oils contained ceramic tracers (0.3 kg/m^3, 150 micron diameter) to permit visualization of fluid flow patterns.

Once the liquid bridges were created, various stimuli were to be imposed on the system. These stimuli included (1) axial oscillation, (2) single disk rotation, (3) disk counter rotation, (4) disk isorotation, (5) disk axial misalignment with isorotation, (6) bridge rupture (by stretching or liquid removal while stretching), and (7) after rupture, merging of the two ruptured drops. Communication between the Payload Specialist and Principal Investigator was realized during portions of the study.

Reportedly, "...when trying to fill the zone, the silicone oil did not get anchored at the disc edge but overflowed it." (1, p. 32) Therefore, the planned experimental procedure (see Ref. (1)) was abandoned and an investigation to find the cause of the anomaly was conducted. <Note: Similar liquid spreading difficulties occurred during another investigation on Spacelab (see

Haynes, Spacelab 1 (Chapter 12)).>

During the redefined experiment, it was determined that the spreading wave, caused by the automated injection of the silicone oil, was large enough to overcome the anti-spreading barrier and jump over the disk edge. Therefore, different injection rates (manually) were attempted. It was determined that "Slowly growing the drop, a maximum liquid angle at the edge was found around 70^{o} to 80^{o}, beyond which the oil swiftly overflowed (with the nominal injection rate [0.8 cm^3/s, 40 mm dia. disk, and 6 mm dia. feed tube], the spreading wave jumped over the edge with a smaller angle; some 20^{o} or 30^{o})." (1, p. 33)

Other experimental parameters were adjusted during the mission (see Reference (1) for details), which included changing the disk size and disk material.

Some meaningful results were obtained by (1) observing the experiment while it was in progress (by the Payload Specialist onboard Spacelab 1 and Principal Investigator on the ground) and (2) post-flight examination of the recorded film. It was reported that:

(1) More attention must be paid to inertial forces present during fluid injection and bridge formation.

(2) Selection of experimental materials (e.g., liquid, disks, cleaning aids) requires a large amount of ground testing.

(3) There were no observable differences in wetting behavior related to the selected disk material (aluminum and stainless steel) or liquid viscosity.

(4) The ceramic tracers were not visible in the more viscous oil but were highly visible in the thinner oil. It was suspected that in the more viscous oil, the tracers may have stuck to the walls of the reservoir and thus, were not visible.

(5) Real-time feedback (via the TV signal) between the ground and Spacelab 1 scientists proved very useful. However, voice communications were hampered: "...pressure on the system prevented detailed scientific discussions with the crew, which were continuously interrupted for long messages and troubleshooting other facilities." (1, p. 36)

(6) "Absence of time correlation in film, poor visualization and the script deficiencies associated with the too many changes needed to recover from failure to get the interface anchored, hinder a deeper analysis of this... experiment." (1, p. 36)

<Note: References (13)-(15) were not available at the time this experiment summary was written.>

Key Words: Capillarity, Liquid Bridges, Liquid Bridge Stability, Liquid Stability, Hydrodynamics, Liquid Injection, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Reservoir, Drops, Meniscus Shape, Meniscus Stability, Free Surface, Surface Tension, Viscosity, Solid/Liquid Interface, Liquid/Gas Interface, Wetting, Wetting Kinetics, Liquid Spreading, Contact Angle, Tracer Particles, Containerless Processing Applications, Photographic Difficulties, Processing Difficulties, Plateau Setup

Number of Samples: Unclear. <Note: Although the Principal Investigator reported that "Two liquid reservoirs" were available, it is not clear how many bridges were actually created using the fluid from these two reservoirs during the course of the experiment.>
Sample Materials: Silicone oil (viscosity: 5×10^{-6} m^2)/s and 100 m^2/sec) with ceramic microsphere solid tracers (0.3 kg m^{-3}, 150 micrometers in diameter)
Container Materials: two stainless steel disks, two aluminum disks

Experiment/Material Applications:
Refer to Martínez, TEXUS 10.

References/Applicable Publications:
(1) Martínez, I.: Liquid Column Stability Experiment 1 ES-331. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 31-36.

(2) Chassay, R. P and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication. (acceleration measurements on Spacelab 1)

(3) Gonafalone, A.: The Fluid Physics Module-A Technical Description. In ESA 5th European Symposium on Materials Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 3-7.

(4) Cerometti, G.: The Fluid Physics Module Design. XXV Rassegna Internazionale Electronica Nucleare Ed Aerospaziale, Rome, March 10-19, 1978, pp. 76-83. (prior to Spacelab 1)

(5) Martínez, I., Haynes, J. M., and Langbein, D.: Fluid Statics and Capillarity. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer Verlag, 1987, pp. 53-80. (related topics)

(6) Da Riva, I. and Ruesga, J. M.: Fluid Physics Module Experiments. Materials Science in Space, ESA SP-114, 1976, pp. 265-275.

(7) Martínez, I.: Floating Zone Under Reduced Gravity, Axisymmetric Equilibrium Shapes. Materials Science in Space, ESA SP-114, 1976, pp. 277-282.

(8) Martínez, I. and Rivas, D.: Plateau Tank Facility for Simulation of Spacelab Experiments. Acta Astronautica, 9, 1982, pp. 339-342.

(9) Meseguer, J.: The Influence of Axial Microgravity on the Breakage of Axisymmetric Slender Liquid Bridges. Journal of Crystal Growth, 62, 1983, pp. 245-250.

(10) Meseguer, J., Mayo, L. A., Llorente, J. C., and Fernandez, A.: Experiments with Liquid Bridges in Simulated Microgravity. Journal of Crystal Growth, 73, 1985, pp. 609-621.

(11) Input received from Principal Investigator I. Da Riva, June 1988.

(12) Input received from Principal Investigator I. Martínez, June 1993.

(13) Da Riva, I. and Martínez, I.: Floating Zone Stability (Exp. 1-ES-331). Material Science in Space, ESA SP-142, pp. 67-73, 1979. (preflight)

(14) Martínez, I.: Zonas líquidas flotantes. Revisita de Ingenieria Aeronàutica y Astronàutica, 165, pp. 19-17, 1977.

(15) Martínez, I. and Sanz, A.: Experiments with Long Liquid Columns Under Microgravity. Proc. of the Seventh European Symposium on Materials and Fluid Sciences in Microgravity, ESA SP-295, 1990, pp. 413-419.

<Note: Many other publications have been written by the investigators which describe research related to this Spacelab 1 experiment.>

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Experiment Origin: Spain
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Fluid Physics Module (FPM) (Same facility as the FPM of Spacelab 1 but with improvements)
Builder of Processing Facility: FIAT C. R., Italy

Experiment:
Floating Liquid Zone (FLIZ) Hydrodynamics, A study of liquid bridges in microgravity (WL-FPM-04)

This Spacelab D1 experiment was the fourth in a series of investigations designed by Da Riva and/or Martínez et al. to study the formation and stability of a liquid bridge under low-gravity conditions (see Da Riva, Spacelab 1; Martínez, TEXUS 10, TEXUS 12 (all in this chapter)). The specific objective of the experiment was to examine the stability of liquid columns subjected to mechanical stimulation.

The experiment was performed in the Spacelab D1 Fluid Physics Module. The module was configured with two aluminum coaxial disks between which a liquid bridge could be created. Each disk had a radius of 0.0175 m and was made with a 30^{o} receding sharp edge to prevent liquid spreading.

A total of six experimental runs were performed during the allotted time. (Reference (2) contains details of each experimental run.) During a run, silicone oil was injected through a hole in the Front Disk (FD). After zone establishment, the liquid bridge was subjected to vibration, rotation of both disks, and/or stretching. Axial vibrations of the liquid column were transmitted via the Rear Disk (RD). Data from the experiment included (1) photographic film, (2) comments from the Payload Specialist, and (3) real-time voice/video downlink (available during portions of the experiment).

After the flight, it was reported that liquid columns subjected to mechanical stimulation were successfully stabilized. Various vibrational frequencies and rotations were applied to the liquid

columns, resulting in the following conclusions:

(1) A large volume (mass) of liquid was well anchored to the sharp edges of the disks during the experiment.

(2) Long cylindrical columns with length/diameter ratios of 2.86 were established several times. The theoretical length/diameter limit for zero-gravity conditions was pi (3.14).

(3) A long cylindrical column was subjected to vibrational frequencies of 0.1, 0.3, 0.7, 1.1, and 1.6 Hz. No movement of the liquid was observed for 0.1 Hz vibration. However, standing waves with 2, 3, 4, and 5 inner nodes were found for the remaining frequencies, respectively. The number of nodes for the respective frequency was successfully predicted by theory.

(4) Destabilization of the columns, caused by rotation at increasing rates, occurred near the theoretical limit.

(5) When liquid bridges were subjected to perturbations beyond the stability limits, the bridges broke and two separate drops formed. The relative volumes of these drops were predicted by theory.

(6) A maximum liquid bridge length of 100 mm (diameter = 35 mm) was achieved during the Spacelab D1 mission. This maximum length appeared to be bounded by the ambient noise (g-jitter) during the mission.

Key Words: Capillarity, Liquid Bridges, Liquid Columns, Liquid Injection, Liquid Expulsion Through a Small Orifice, Liquid Reservoir, Liquid Bridge Stability, Liquid Column Rupture, Hydrodynamics, Meniscus Shape, Meniscus Vibration, Meniscus Stability, Axial Oscillation, Standing Waves, Free Surface, Liquid/Gas Interface, Solid/Liquid Interface, Rotating Fluids, Sample Rotation, Liquid Vibration, Liquid Dynamic Response, Drops, Wetting, Wetting Kinetics, Liquid Spreading, Acceleration Effects, Tracer Particles, Containerless Processing Applications

Number of Samples: One experimental setup was used to realize six experimental runs.
Sample Materials: Dimethyl silicone oil, viscosity: $5 \times 10^{-6} m^2/s$, density: 920 kg m^{-3}, surface tension: 0.02 N m^{-1}. The oil contained tracers (Eccospheres, 0.15×10^{-3}m diameter, 0.1 kg m^{-3} concentration)
Container Materials: two aluminum disks

Experiment/Material Applications:
See Martínez, TEXUS 10.

References/Applicable Publications:
(1) Da Riva, I. and Martínez, I.: Floating Liquid Zones. Natur wissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 345-347. (post-flight)

(2) Martínez, I. and Meseguer, J.: Floating Liquid Zones in Microgravity. In BMFT/DFVLR Scientific Results of the Germany Spacelab Mission D1, Norderney Symposium, Germany, August 27-29, 1986, pp. 105-112.

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(5) Sanz, A. and Martínez, I.: Minimum Volume for a Liquid Bridge Between Two Disks. J. Colloid Interface Sci., 93 (1983), pp. 235-240. (no D1 results, yet related)

(6) Martínez, I.: Stability of Liquid Bridges. Results of SL-D1 Experiment, Acta Astronautica, 8, 1987, pp. 449-453.

(7) Meseguer, J.: The Breaking of Axisymmetric Slender Liquid Bridges. Journal of Fluid Mechanics, 130, 1983, pp. 123-151.

(8) Gonfalone, A.: The Fluid Physics Module-A Technical Description. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 3-7. (status post-Spacelab 1, but prior to D1.)

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(15) Input received from Principal Investigator I. Martínez, June 1993.

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Co-Investigator(s): Unknown
Affiliation(s): (1) Kodak Limited, Harrow, United Kingdom

Experiment Origin: United Kingdom
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Fluid Physics Module (FPM)
Builder of Processing Facility: FIAT C. R., Italy

Experiment:
Capillary Forces 1ES329

When a liquid contacts a solid surface in the presence of a second fluid, non-wetting, wetting, or spreading of the liquid on the surface must occur. (An example of such a solid/2-fluid system is a liquid zone which touches a circular end plate in the presence of the surrounding air.) If spreading occurs, the bulk liquid remains in contact with a thin film (of the bulk fluid) on the solid. Throughout this contact, gravity dependent "...draining forces balance the forces of adhesion between the solid and the liquid to produce... [the] thin film." (1, p. 9)

This experiment was the first in a series of low-gravity investigations designed by Padday to study the intermolecular forces between contacting solids and liquids. The specific objectives of the experiment were to (1) study the capillary properties of a silicone oil liquid zone held axisymetrically between two end plates, (2) investigate (in particular) the low-gravity formation and properties of the thin films forming between the contacting solid and liquid and (3) examine the stability of the zone.

The Spacelab Fluid Physics Module was configured such that a large liquid zone of silicone oil could be suspended between (1) a 10 cm diameter titanium plate shaped as a shallow cone, and (2) a 3 cm diameter plate of aluminum. (The outer edge of the titanium plate had been treated with an anti-wetting agent.) It was noted that "By adjusting the separation and the volume of the liquid, conditions may be obtained whereby an outer annulus of the cone plate holds a spread film of liquid in equilibrium with the liquid zone. Thus the negative capillary pressure balances the positive disjoining pressure of the thin film." (1, p. 10)

It was further noted that in the low-gravity environment, "Very low capillary pressures can be created which allow the corresponding spread film to become... much thicker than is possible

on earth." (1, p. 9)

Prior to the flight, Laplace's capillary equation was used to calculate the theoretical shapes and stability properties of the axisymmetric zones. One interesting fact that the analysis indicated was that a spreading film would form if "...the angle of contact of the zone with the large end plate reached the cone angle before the radius limit of the outer edge of the plate." (1, p. 10) (Further details concerning the analysis were presented in Reference (1).)

It appears that the liquid zones were created during the mission by injecting silicone oil through a hole in the titanium plate towards the smaller aluminum plate. A 16 mm Vinten Camera Mark B was used to record the resulting liquid zone shape and film thickness. During the two experimental runs, voice contact between the ground investigative crew and the payload specialist operating the equipment was available.

During the first experiment run "...the silicon oil overspread the small end plate and wetted completely a plastic backing plate of 3 cm radius (situated immediately behind the 1.5 cm radius aluminum plate). Examination of the photographic record... showed that the plate was wetted very quickly and that either due to inertia within the liquid or to imposed wetting movement, the 28[degree] advancing contact angle of silicon oil on the antispread was exceeded and unwanted wetting took place.... [I]t is abundantly clear that the overwetting was caused by operations and not by antispread failure." (1, p. 11) After the anomaly, "new settings for the zone" (presumably new injection procedures) were uplinked to the crewman operating the experiment. <Note: It appears to the editors that during the remainder of the run, six different liquid zones were examined.> Each of these zones had a slightly different separation length and zone volume. The shapes of these zones were compared to the predicted theoretical shapes. For all of these six Spacelab zones, a well-defined wetting film appears to have been formed. Further, the liquid zones were also well defined and had a Laplace shape. It was reported that during the examination of one of the zones "...a 100 v D. C. potential was applied between the end plates." (1, p. 12) A brief note in the introductory abstract of Reference (1) indicated that the "electrical field showed no effects." It was also reported that "During the 20 second lapse of each experiment no measurable perturbations to zone shape, contact angle or film thickness was detected." (1, p. 12)

Reference (1) indicated that some of the data had not yet been analyzed for three of the more interesting zones. Reference (1) did report that the capillary pressures of these three zones were -0.34, -0.24, and -0.40 N/m^2. It was further reported that al-

though these pressures were small as expected, they "...were still too large to obtain films of sufficient thickness." (1, p. 12)

Because of the experience gained during the first run, the second experimental run was much more successful. "The small end plate had been cleaned and the edges retreated with antispread material during the flight." (1, p. 12) During this second run, nine different liquid zones were examined. Each of these zones had a slightly different plate separation length and zone volume. (The capillary pressures of the zones were between -0.08 and -0.009 N/m^2, the disjoining pressures were between 0.01 and 0.08 N/m^2.) One of the zones created during the later part of the run had a well defined film of up to 0.2 mm thick (capillary pressure -0.72 N/m^2). It was reported that such zones with thicker films did not correlate as well to the predicted shapes.

"A further feature of some... [of the zones created during Run 2] was that a thickening of the zone itself was detected in the region where the liquid zone approached the wetting film." (1, p. 13)

During the last portion of the second experimental run, a large liquid bridge was broken at the expected separation distance. The rupture of this Spacelab bridge occurred much more rapidly (0.25 to 0.3 seconds) than did rupture of a correspondingly sized bridge created on Earth in a Plateau tank (several seconds).

Post-flight analysis of the exposed film (see Reference (1) for details) led to the following conclusions:
(1) The disjointing pressures achieved under low-gravity conditions were much lower than possible on Earth.
(2) The thickness of the liquid film under low-gravity conditions was much greater than expected.
(3) At lower capillary pressures, very thick liquid films were obtained. At higher pressures, the films were thin.
(4) The breakage of the liquid bridge (second experimental run) was much faster than observed on Earth. However, the separation distance at breakage was as expected.

Key Words: Capillarity, Capillary Forces, Capillary Pressure, Meniscus Shape, Meniscus Stability, Liquid Bridges, Liquid Columns, Liquid Bridge Stability, Liquid Column Rupture, Solid/Liquid Interface, Liquid/Gas Interface, Liquid Films, Thin Films, Free Surface, Surface Tension, Wetting, Wetting Kinetics, Liquid Spreading, Contact Angle, Electric Field, Liquid Expulsion Through a Small Orifice, Liquid Injection, Coated Surfaces, Plateau Setup, Processing Difficulties

Number of Samples: One liquid zone setup, allowing the creation of 15 different liquid zones.
Sample Materials: silicone oil, dimethyl siloxane, Dow Corning d.c. 200/5 cs
Container Materials: endplates: titanium; aluminum
The employed anti-spreading material was developed at Kodak Laboratories, applied as an aqueous solution and dried.
(Ti*, Al*)

Experiment/Material Applications:
No discussion of the material application could be located in the published literature. However, the research is probably most applicable to (although not limited to) liquid zones created during float zone crystal growth.

References/Applicable Publications:
(1) Padday, J. F.: Capillary Forces in a Low Gravity Environment. In ESA 5th European Symposium Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 9-14. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication. (acceleration measurements on Spacelab 1)

(3) Padday, J. F.: The Behaviour and Management of Liquid Systems in Low Gravity. In Proc. of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 49-56. (related research; post-flight)

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Experiment Origin: United Kingdom
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Fluid Physics Module (FPM) (same facility as the FPM of Spacelab 1 but with improvements)
Builder of Processing Facility: Fiat C. R., Italy

Experiment:
Capillary Experiments- Adhesion Forces in Liquid Films (WL-FPM 06)

This Spacelab D1 experiment was the second in a series of low-gravity investigations designed by Padday to study intermolecular forces between solids and liquids (see Padday, Spacelab 1).

During the earlier Spacelab 1 experiment, "...it was discovered that very low capillary pressures of a zone in contact with a liquid film were achieved. At these low pressures ($0.01\ N/m...^2$) the corresponding wetting layer appeared to be excessively thick, that is up to 0.2 mm. However the scarce data and the associated problems in proving equilibrium led to the need to obtain much more and better refined data in a second flight experiment." (2, p. 251)

The objectives of this study were to (1) study the capillary properties of a silicone oil liquid zone held axisymetrically between two end plates, (2) investigate the low gravity formation and properties of the thin films (at even lower capillary pressures than had been realized on Spacelab 1) which form between the contacting solid (titanium plate) and liquid (silicone oil), (3) create and investigate wetting layers which were in equilibrium with axisymmetric Laplace zones, (4) investigate vibration and rotation effects on Laplace zones and wetting layers, and (5) quantitatively confirm the stability increase of a liquid zone caused by vibration.

"The theoretical shapes of zones at equilibrium between endplates... [were] derived from numerical solutions of Laplace's capillary equation.... For each combination of [zone] volume and [plate] separation, there is usually two Laplace shapes, one of which is stable and the other unstable. A further small group

are those with a unique shape and these are the ones reached when a zone is critically stable (i.e. the two points merge) and the zone breaks." (2, p. 252)

During the Spacelab D1 mission, liquid zones were created between two axisymmetric end plates. "The large end plate... [was] cone shaped with a diameter of 100 mm and a cone angle of 10[degrees] and was made from a single billet of titanium. Unlike the... [Spacelab 1] end plate this one was made with sharp edges and all non wetted surfaces were treated with a layer of suitable antispread material. This cone endplate was mounted at the rear end of the Fluid Physics Module.... The small endplate, mounted at th [sic] front end, was fed with liquid through a rosette of five small hole [sic] in its surface connecting to a syringe which had a delivery accuracy of + or - 0.02 cc.

"The small end plate was flat, of 30 mm diameter and made of aluminum. The edges of the wetted surfaces were again sharp and the rear side and supporting surfaces treated with antispread coating. Silicone oil (viscosity 5cs) was used for all experiments and no tracers were added. The density of the silicone oil was given as 0.93 g/cc and surface tension as 10 mN/m." (2, p. 252)

The experiment runs were photographed using a 16 mm Vinten camera. The camera usually operated in single frame mode except when the liquid began to rupture. At the initiation of rupture, the camera instantly changed to cine mode.

The experimental sequence included (1) "...adjusting the volume of silicone oil and the distance of separation so that a predetermined series of shapes with and without wetting films were produced, hopefully at stable equilibrium...." (2, p. 253), (2) later rotating the cone plate alone, or both endplates at 5 rpm (rotation of the cone plate or both plates was not performed for all the zones), and (3) vibrating the cone plate at an amplitude of 0.2 mm and a frequency of 1.9 Hz.

Three experimental conditions were examined during this study: (1) liquid zones which were very nearly catenoid shaped and without a wetting film or stimulation (to study liquid zone properties), (2) liquid zones with a wetting film on the cone plate surface (to study properties of wetting films), and (3) liquid zones stimulated by rotation and axial vibration (to study stability of liquid zones). The results from the above conditions are reported below:

Near Catenoid Zones:

During the first experimental run, five zones were created. Each of these zones had a slightly different liquid volume and plate separation distance. "The settings were arranged so that all shapes would be stable and have the shape of "perfect" catenoids...." (2, p. 253) (The capillary pressures of these near catenoid zones ranged between -0.013 and +0.032 N/m^2.) One of the experimental zone shapes was compared to the expected theoretical shape. "It is seen that there is excellent agreement between the experimental and theoretical data indicating that the methods of measurement and of data analysis were working satisfactorily." (2, p. 253) During the run, one of the five zones broke. "...the zone was broken... first at constant separation by reducing the zone volume and then, after repairing the zone, by stretching the zone at constant volume. Within experimental error, the zone broke at the theoretical limit in each experiment." (2, p. 253)

Properties of Wetting Layers:

"It was found that when zones were created with small volumes and small separation distances, the liquid partitioned itself between the Laplace zone and a newly formed wetting layer on the cone's surface. This wetting layer was caused by the angle of contact of the liquid at the cone's surface, reaching its limiting value. In this case the liquid did not recede from the cone's surface and so the angle reached was assumed to be that of the cone (10 deg.). (2, p. 254) Reportedly, due to flight delays, only two data points for this part of the study could be obtained. <Note: More details of the wetting film results are included in Reference (2).>

Rotational and Vibrational Stimulation of Zones:

The first run of this portion of the experiment yielded three points on the stability diagram. However, "...the repeat of these points and the addition of [a second run data points] were unrecorded because of major camera failure. The precise separation distances at rupture, determined under stimulation, were recorded both manually and on the data logging system thereby saving the essential part of the experiment." (2, p. 254) It was reported that both monorotation (rotation of cone plate only) and isorotation (rotation of both end plates) did not visibly change the zone shape. However, theoretical calculations (Laplace equation) did predict a slight shape change. Induced axial vibration did not induce harmonic wave movement and apparently *increased* the stability of the zone.

Rupture of the liquid zones was also analyzed during these experiments. It was reported that rupture occurs in four steps:

(1) A series of unstable equilibrium Laplace shapes with continuously decreasing neck size.

(2) Liquid cascading through a series of non-Laplace shapes (still axisymmetric) with liquid at each end plate being pyramidal and connected by a thin column.

(3) Rupture occurring rapidly at two places in the thin column. The column becomes a satellite drop and liquid at end plates relaxes to spherical drop shape.

(4) Satellite rebounds between liquid/air surfaces, but not penetrating these surfaces.

Key Words: Capillarity, Capillary Forces, Capillary Pressure, Hydrodynamics, Meniscus Shape, Meniscus Stability, Liquid Bridges, Liquid Columns, Liquid Column Rupture, Liquid Bridge Stability, Rotating Fluids, Sample Rotation, Liquid Vibration, Liquid Dynamic Response, Axial Oscillations, Sample Deformation, Sample Necking, Solid/Liquid Interface, Liquid/Gas Interface, Thin Films, Liquid Films, Free Surface, Wetting Kinetics, Liquid Spreading, Contact Angle, Liquid Expulsion Through a Small Orifice, Liquid Injection, Coated Surfaces, Photographic Difficulties

Number of Samples: not applicable
Sample Materials: silicone oil, (viscosity 5 cs) no tracers, density = 0.93 g/cc, surface tension = 19 mN/m
Container Materials: Zone endplate surfaces: aluminum, titanium. Both surfaces were coated with an antispread material. (Al*, Ti*)

Experiment/Material Applications:
"Photographic materials are manufactured terrestrially by spreading layers of liquid (photographic emulsion) onto a flexibly solid support. The interest in this type of experiment is to obtain scientific data on the properties of liquid layers that have not drained to their terrestrial equilibrium thickness. Their formation is possible only in a low gravity environment." (1, p. 42)

References/Applicable Publications:

(1) Padday, J. F.: Adhesion Forces in Liquid Films. In Scientific Goals - D1 Spacelab Mission, Eds. Jansen and Sahm, Giesseri Inst. RWTH, Aachen, D-5000 Aachen, 1985, pp. 42-44. (preflight)

(2) Padday, J. F.: Capillary Forces in Low Gravity. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA-256, pp. 251-256. (post-flight)

(3) Padday, J. F.: The Behaviour and Management of Liquid Systems in Low Gravity. In Proc. of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 49-56. (related research; post-flight)

(4) Langbein, D.: Fluid Physics. In Scientific Results of the German Spacelab Mission, Norderney Symposium, August 27-29, 1986, pp. 93-104. (post-flight; discusses other fluids experiments)

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Experiment Origin: USA and possibly Japan
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-004
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of this experiment within G-004: Utah State University, Logan, Utah
Processing Facility: Container with agitator on one end
Builder of Processing Facility: Unknown

Experiment:
Capillary Wave Study

This experiment was one of six investigations housed within the G-004 Get Away Special Canister during STS-011. Three other experiments (of the six) were applicable to this data base (see Thomas, S., STS-011 (Chapter 12); and Gadsden, STS-011 (two experiments, Chapters 15 and 18).

The experiment was the first in a series of shuttle experiments designed by Kitaura et al. to study liquid capillary waves. "The waves are driven by surface tension forces which are dominant in a microgravity environment." (3, p. 8; also 4, p. 27)

Very little information appears to have been published which describes the objectives, setup, and results of this investigation. A document released prior to the shuttle launch briefly detailed the experiment goals and configuration: "The... experiment will look at capillary waves on water surfaces. The experiment consists of a small water filled chamber with a small agitator at one end. The surface will be forced to form by including a wettable surface over part of the chamber and a hydrophobic surface over the remainder. The wave patterns will be photographed using an 8 mm movie camera." (1, pp. 2-5)

Reference (7) appears to have the only summary of the experimental results. Reportedly, the experiment failed "due to weak battery capacities." It was suspected that "...the batteries degraded during the long wait in the orbiter before launch." (7, p. 28)

No further information concerning the experiment appears to be available.

Key Words: Capillarity, Capillary Waves, Hydrodynamics, Free Surface, Surface Tension, Wetting, Wetting of Container, Non-Wetting of Container, Hydrophobic Surfaces, Coated Surfaces, Liquid/Gas Interface, Solid/Liquid Interface, Contained Fluids, Liquid Reservoir, Liquid Dynamic Response, Processing Difficulties, Battery Drain

Number of Samples: one experiment cell
Sample Materials: water
Container Materials: unknown

Experiment/Material Applications:
See Kitaura, STS-017.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS-11. JSC-17645 Annex STS-11, December 2, 1983. (preflight; very short description)

(2) Student GAS Program Internal Documentation, Utah State University, Logan, UT 84322, 1984.

(3) Get Away Special (GAS) Payloads (STS-11). In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, Published by the Engineering Directorate, p. 8. (very short description)

(4) 41-B Tenth Space Shuttle Mission Press Kit, February 1984, p. 27. (preflight)

(5) STS-11 Get Away Special Payload Description, NASA News, NASA GSFC, 1984.

(6) NASA Press Kit, STS-12, p. 23. (preflight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Getaway Special Canister mission history)

(8) Input received from Principal Investigator T. Kitaura, July 1993.

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Experiment Origin: USA and possibly Japan
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-518
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-518: Utah State University, Logan, Utah
Processing Facility: Container with agitator on one end
Builder of Processing Facility: Unknown

Experiment:
Capillary Wave Study

This experiment was one of four investigations housed within the G-518 Get Away Special (GAS) canister during STS-017. Three other experiments (of the four) were applicable to this data base (see Thomas, S., STS-017 (Chapter 12; Walden, STS-017 (Chapter 18); "Solder Flux Separation," STS-017 (Principal Investigator unknown, Chapter 18)).

The experiment was the second in a series of experiments designed by Kitaura et al. to study liquid capillary waves (see Kitaura, STS-011). "Under zero-g, the restoring force in wave action is surface tension." (6, p. 23)

Very little information appears to have been published which describes the objectives, setup and results of this investigation. A document released prior to the shuttle launch briefly detailed the expected experiment goals and configuration: "The experiment consists of a chamber containing water. A surface will be formed by the use of two appropriately shaped walls made of two materials with widely differing affinities for water.

"After the experiment is turned on the water will be injected into the chamber and the surface of the water will be agitated.

An 8 mm camera in the single frame mode will photograph the disturbances. A small fluorescent light operated on six volts will provide light for the camera." (7)

The description of the experiment in the Cargo Systems Manual for STS-017 (41-G) was nearly an exact duplicate of that provided for the earlier mission and detailed under Kitaura, STS-011.

Reference (3) appears to contain the only summary of the experimental results. Reportedly, the experiment "failed to activate". The equipment functioned "...fine after return to earth. Two more months of testing couldn't duplicate the symptoms." (3, p. 32)

No further information concerning this experiment appears to be available at this time.

Key Words: Capillarity, Capillary Waves, Hydrodynamics, Free Surface, Surface Tension, Wetting, Wetting of Container, Hydrophobic Surfaces, Coated Surfaces, Liquid/Gas Interface, Solid/Liquid Interface, Contained Fluids, Liquid Dynamic Response, Liquid Injection, Liquid Reservoir, Processing Difficulties

Number of Samples: one experiment cell
Sample Materials: water
Container Materials: unknown

Experiment/Material Applications:
This research was expected to yield new information on the nature of capillary waves.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS 41-G. JSC-17645 41-G, September 4, 1984. (short description; preflight)

(2) NASA Space Shuttle Mission 41-G Press Kit, October 1984, pp. 24-25. (preflight)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Getaway Special Canister mission history)

(4) G-518 Payload Accommodations Requirements, NASA Goddard Space Flight Center, March 20, 1984.

(5) Press Release for G-518, Utah State University, Logan Utah, 1984.

(6) NASA Press Kit, STS-12, p. 23. (preflight)

(7) Information provided by Globesat Incorporated, Logan Utah. (preflight)

(8) Input received from Principal Investigator T. Kitaura, July 1993.

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Experiment Origin: Spain
Mission: TEXUS 10
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-9: Liquid Column Cell (LCC)
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Maximum Injection Rate in a Floating Zone

This TEXUS 10 experiment was the second in a series of investigations designed by Martínez and/or Da Riva et al. to study the formation and stability of a liquid bridge under low-gravity conditions (see Da Riva, Spacelab 1 (this chapter)). The primary goal of the experiment was to establish a long, cylindrical column of fluid in a minimum amount of time.

The TEXUS 10 experiment was performed in the TEM 06-9 Liquid Column Cell. The apparatus contained two aluminum disks between which a silicone oil bridge (zone) could be created. Each disk was 30 mm in diameter with its lateral sides (5 mm) cut back to 45^{o} to increase its anchoring capability.

Because fluid spillage while attempting to create a liquid bridge is irreversible, a smooth, progressive, bridge-building approach was planned. The zones were to be established by the injection of liquid through one disk. The maximum zone length achievable was 82 mm, and the length could be built up at ten different speeds (by adjusting axial separation of disks and simultaneous injection of fluid). The build-up rate could be increased from 2 mm/s to 20 mm/s at increments of 2 mm/s.

During the mission, a malfunction of the apparatus prevented the experiment from starting: "...a mechanical problem in the drive motor prevented it from functioning properly...." (1, p. 324). Reportedly, because of this malfunction, no liquid could be deployed and thus no results could be obtained. The experiment was repeated on TEXUS 12.

No other information concerning this experiment could be located.

Key Words: Capillarity, Liquid Bridges, Liquid Columns, Liquid Bridge Stability, Hydrodynamics, Liquid/Gas Interface, Solid/Liquid Interface, Free Surface, Surface Tension, Liquid Injection, Maximum Liquid Injection Rate, Liquid Transfer, Liquid Reservoir, Flow Rates, Liquid Expulsion Through a Small Orifice, Wetting, Liquid Spreading, Containerless Processing Applications, Hardware Malfunction, Sample Not Processed As Planned

Number of Samples: One liquid bridge setup. Several liquid bridges were to be created.
Sample Materials: dimethyl-silicone oil
Container Materials: aluminum disks
(Al*)

Experiment/Material Applications:
Understanding the formation of liquid bridges is important during the float-zone processing of crystals.

The specific objective of this TEXUS 10 experiment was to quickly create a liquid bridge in the reduced gravity environment.

References/Applicable Publications:
(1) Martínez, I. and Sanz, A.: Long Liquid Bridges Aboard Sounding Rockets. ESA Journal, Vol. 9, No. 3, 1985, pp. 323-328. (TEXUS 12 results only, mentions malfunction occurred in TEXUS 10 Experiment)

(2) Input received from Experiment Investigators, June 1988 and September, 1988.

(3) Input received from Principal Investigator I. Martínez, June 1993.

(4) Sanz, A. and Perales, J. M.: Liquid Bridge Formation. Appl. Microgravity Tech., 2(3), pp. 133-141, 1989.

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Co-Investigator(s): None
Affiliation(s): (1,2) Universidad Politécnica, E.T.S.I. Aeronáuticos, Madrid, Spain

Experiment Origin: Spain
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-9: Liquid Column Cell (LCC) (Developed for TEXUS 10 and then modified for TEXUS 12)
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Maximum Injection Rate in a Floating Zone

Understanding the parameters associated with the formation and stability of liquid bridges can benefit a variety of disciplines (e.g., crystal growth of silicon using a molten bridge configuration). Liquid bridge research opportunities during long-term, low-gravity experimental periods are limited however, so short-term sounding rocket experiments become a secondary testing alternative. The short amount of time that the experiment is under low-gravity conditions (approximately 6 minutes) requires that the liquid bridge be formed as quickly as possible. If the bridge can be created quickly, sufficient time would be available for meaningful investigation.

This TEXUS 12 experiment was the third in a series of investigations designed by Martínez and/or Da Riva et al. to study the formation and stability of a liquid bridge under low-gravity conditions (see Da Riva, Spacelab 1; Martínez, TEXUS 10 (both in this chapter)). The primary objectives of the study were to (1) establish a liquid bridge in a minimum amount of time and (2) observe several bridge properties. A secondary objective of the experiment was to test experimental concepts to be used during future STS Spacelab mission experiments.

The TEXUS 12 experiment was performed in the TEM 06-9 Liquid Column Cell. The apparatus contained two aluminum disks between which a silicone oil bridge (zone) was created. Each disk was 30 mm in diameter with its lateral sides (5 mm) cut back to 45^{o} to increase its anchoring capability. The zones were established by the injection of liquid through one disk. The maximum zone length achievable was 82 mm, and the length could be built up at ten different speeds (by adjusting axial separation of disks and simultaneous injection of fluid). The build-up rate could be in-

creased from 2 mm/s to 20 mm/s at increments of 2 mm/s. Observation of the zone was possible by an 80 x 120 mm lit field equipped with a millimeter rastar. The experimental process was filmed using a 16 mm cine camera at 20 frames/sec.

Three "stretchings" of liquid bridge zones were achieved during the rocket experiment. During each of these stretchings (1) the liquid bridge was expanded from 4 mm to 82 mm and (2) the bridge formation speed was either 2 mm/s (stretch #1), 4 mm/s (stretch #2) or 6 mm/s (stretch #3).

It was reported that the column behaved as expected during the first stretching with a perfectly cylindrical shape. A slight necking occurred near the injection disk at the end of its formation. This necking led to slow oscillations of the column with a frequency of approximately 10 seconds.

The second column also was very cylindrical up to about an 80 mm length (stretched at 4 mm/s). At this point a neck occurred on the fixed disk side with a period of 12 seconds. "At the end, larger reflecting waves than before [the first column] could be observed. In both cases the oscillation period at maximum separation is close to the theoretical prediction of 10.3 seconds." (7, p. 27)

"Early on the third stretching (injection disk speed 6 mm/s), the column developed a permanent necking (about 30% of radial deformation peak to peak) which, surprisingly, was located close to the fixed disk. This is contrary to the ground test experience. The undulation continued after stopping the injection...and gave way to the disruption of the liquid bridge...." (7, p. 28) <Note: the maximum zone length achieved was 82 mm.>

Post-flight examination of the data revealed "...that large liquid masses can be freed from a reservoir and accurately positioned between two solid coaxial disks, all in less than a minute in weightlessness, thus enlarging the range of possible experiments with liquid bridges aboard sounding rockets." (1, p. 326) It was also reported that inertial forces significantly affected the behavior of the bridges.

Key Words: Capillarity, Liquid Bridges, Liquid Columns, Liquid Bridge Stability, Liquid Column Rupture, Meniscus Shape, Meniscus Vibration, Meniscus Stability, Sample Necking, Sample Deformation, Fluid Oscillation, Hydrodynamics, Liquid Dynamic Response, Solid/Liquid Interface, Liquid/Gas Interface, Free Surface, Surface Tension, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Reservoir, Liquid Injection, Liquid Spreading, Wetting, Wetting Kinetics, Maximum Liquid Injection Rate, Flow Rates, Containerless Processing Applications

Number of Samples: One experimental setup was used to create three liquid bridges.
Sample Materials: dimethyl-silicone oil (viscosity = 5×10^{-6} m^2/s, density = 920 kg m^{-3}, surface tension = 0.02 N m^{-1}
Container Materials: Aluminum disks
(Al*)

Experiment/Material Applications:
See above experiment summary and Martínez, TEXUS 10.

References/Applicable Publications:
(1) Martínez, I. and Sanz, A.: Long Liquid Bridges Aboard Sounding Rockets. ESA Journal, Vol. 9, No. 3, 1985, pp. 323-328. (post-flight).

(2) Meseguer, J. and Sanz, A.: One-Dimensional Linear Analysis of the Liquid Injection or Removal in a Liquid Bridge. In Acta Astronautica, Vol. 15, Number 8, 1987, pp. 573-576. (post-flight; theoretical treatment)

(3) Martínez, I. and Sanz, A: Long Liquid Bridges Aboard Sounding Rockets. In TEXUS 11/12 Abschlussbericht, 1985, pp. 69-73. (post-flight)

(4) Input received from Experiment Investigators, June 1988 and September 1988.

(5) Sanz, A. and Perales, J. M.: Liquid Bridge Formation. In Applied Microgravity Technology, Vol. 2, Number 3, 1989, pp. 133-141. (post-flight)

(6) Meseguer, J., Sanz, A., and Perales, J. M.: Axisymmetric Long Liquid Bridges Stability and Resonances. In Applied Microgravity Technology, Vol. 2, Number 4, 1990, pp. 186-192. (post-flight; theoretical treatment of liquid bridge stability)

(7) Long Liquid Bridges Aboard Sounding Rockets. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 26-28. (post-flight)

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Experiment Origin: Spain
Mission: TEXUS 18
Launch Date/Expt. Date: May 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-9: Liquid Column Cell, LCC (developed for TEXUS 10 and modified for TEXUS 12 and TEXUS 18)
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Freezing of a Long Liquid Column

This TEXUS 18 experiment was the fifth in a series of investigations designed by Martínez and/or Da Riva et al. to study the formation and stability of a liquid bridge under low-gravity conditions (see Da Riva, Spacelab 1, Spacelab D1; Martínez, TEXUS 10, TEXUS 12 (all in this chapter)).

The TEXUS 18 experiment was the first in the Martínez/Da Riva investigative series to solidify a liquid bridge. Reportedly, the specific objective of this TEXUS 18 investigation "...was to measure the so called receding contact angle... [gamma]... on the solidification with a free surface." (1, p. 1)

The experiment was performed in the TEXUS TEM 06-9 Liquid Column Cell (LCC). This cell was modified after the TEXUS 12 experiment to include (1) a cooling system, (2) laser illumination, (3) video recording equipment (replacing the cine camera used on TEXUS 12), and (4) eight thermocouples. Three of the eight thermocouples were located in the cooling disk (two of which were used as a heat meter) and five thermocouples were located in the liquid column.

During the first minute of low-gravity conditions, a 86 mm long, 30 mm diameter column of water was established between two aluminum coaxial disks. One disk was then cooled (via evaporation of a halogen carbon compound, R-12) to approximately 210 K. All data (thermal and video) were transmitted to the ground on a real-time basis.

Post-flight examination of the video data revealed that there were problems with the recording. "...the most defeating being that the video transmission seriously deteriorated the image

recording, adding some flickering and image distortions that prevent[ed] any simple, automated, image analysis. Besides, light contrast in the images is much poorer than expected...." (1, p. 6) It was reported that the problems with the recording prevented a detailed contact angle analysis (gamma was near zero value).

Problems with the thermal data also appeared. "..the heat-meter data [was] unreliable (quick-look and final data do not match) and an out of range state was reached. Besides, the cooling law achieved (exemplified by the temperature of the disc) is not simple and not reproducible. An interesting kick can be seen in the plot of the disc temperature at about 260 K, perhaps due to initial undercooling of the liquid and subsequent release of the solidification enthalpy, but the available diagnostics do not allow it to be elucidated. The temperature variations at all thermocouple positions (and especially at the discs) before... cooling started... also have no explanation." (1, p. 6)

Despite these problems, some results were reported. These included:

(1) While it was expected that the thermal gradient near the solidification front (approximately 10 ^{o}C/mm) would produce Marangoni convection and observable movement of tracers within the fluid, the particles were nearly quiescent.

(2) The video images indicated that the solidification front remained planar through the process.

(3) During the re-entry phase of the mission, the bridge was broken and "...the three-phase contact line remain[ed] attached to the corner of the growing crystal." (1, p. 7)

Numerical simulation with a thermal model agreed well with the experimental results "...except that the temperature profiles of the thermocouples readout appear to go down (cool) later and steeper than the onedimensional <sic> thermal model. The effect of lateral heat input from the air atmosphere could explain the first effect but goes against the latter." (1, p. 7) (Details of the theoretical model can be found in Reference (1).)

Key Words: Capillarity, Liquid Bridges, Float Zones, Solidification, Freezing, Liquid Bridge Stability, Liquid Column Rupture, Meniscus Shape, Contact Angle, Surface Tension, Liquid Expulsion Through a Small Orifice, Liquid Injection, Maximum Liquid Injection Rate, Solid/Liquid Interface, Liquid/Gas Interface, Solidification Front Physics, Planar Solidification Interface, Free Surface, Marangoni Convection, Undercooling, Thermal Gradient, Tracer Particles, Containerless Processing Applications, Illumination System, Photographic Difficulties, Illumination Difficulties, Vehicle Re-Entry Forces/Vibration, Hardware Malfunction

Number of Samples: one
Sample Materials: deaeriated, distilled water (with 0.02 kg/m^3 of sodium chloride and ceramic tracers)
Container Materials: aluminum disks
(Al*)

Experiment/Material Applications:
See Martínez, TEXUS 10

Water was selected for this investigation because experimental constraints required that the working fluid (1) be transparent for visualization, (2) have a well-defined melting point, (3) be nonvolatile to avoid liquid volume variations, (4) be compatible with tracers, and (5) be nontoxic and nonflammable.

References/Applicable Publications:
(1) Martínez, I., Sanz, A., Perales, J. M., and Meseguer, J.: Results of a Liquid the Freezing of a Long liquid Column Experiment on TEXUS 18. Submitted to ESA Journal 9/16/88. <Note: The current status of this document is unclear. It appears the document may have been published (with the authors listed above) as: Freezing of a Long Liquid Column on the TEXUS 18 Sounding Rocket Flight. ESA Journal Volume 12 (1988), pp. 483-489.>

(2) Martínez, I. and Eyer, A.: Liquid Bridge Analysis of Silicon Growth Experiments under Microgravity. Journal of Crystal Growth, Vol. 75, 1986, pp. 535-544. (related research)

(3) Sanz, A.: The Crystallization of a Molten Sphere. Journal of Crystal Growth, Vol. 74, 1986, pp. 642-655. (related research)

(4) Sanz, A.: Meseguer, J., and Mayo, L.: The Influence of Gravity in the Solidification of a Drop. Journal of Crystal Growth 82, 1986, pp. 81-88. (related research: Walter, Skylab InSb sphere experiment and Kölker, SL-1 and D1 silicon sphere experiment.)

(5) Input received from Experiment Investigators, June 1988 and September 1988.

(6) Sanz, A. and Perales, J. M.: Liquid Bridge Formation. In Applied Microgravity Technology, Vol. 2, Number 3, 1989, pp. 133-141. (post-flight)

(7) Meseguer, J., Sanz, A., and Perales, J. M.: Axisymmetric Long Liquid Bridges Stability and Resonances. Applied Microgravity Technology, Vol. 2, Number 4, 1990, pp. 186-192. (post-flight; theoretical treatment of liquid bridge stability)

(8) Meseguer, J. and Sanz, A.: One-Dimensional Linear Analysis of the Liquid Injection or Removal in a Liquid Bridge. In Acta Astronautica, Vol. 15, Number 8, 1987, pp. 573-576. (post-flight; theoretical treatment)

(9) Freezing of a Long Liquid Column. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 30-31. (post-flight)

(10) Input received from Principal Investigator I. Martínez, June 1993.

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Co-Investigator(s): Izquierdo, M. (Project Engineer) (2)
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Experiment Origin: USA
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) Canister G-034
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-034: Texas High Schools (Ysleta and El Paso Districts)/Dickshire Coors, El Paso, Texas
Processing Facility: Experimental package configured with wire screen, freon 113, and a capacitor.
Builder of Processing Facility: Unknown

Experiment:
Wicking of Freon

This investigation was one of thirteen experiments housed within the G-034 Get Away Special Canister during the STS-025 mission. (Three other of the thirteen investigations are applicable to this data base; see Casarez, STS-025 (Chapter 18); M. Moore, STS-025 (Chapter 8); Thurston, STS-025 (Chapter 18).) The specific objective of the experiment was to measure the wicking of a simulated fuel (during vehicle acceleration) on a small sample of wire fuel screen.

The sample of screen material used in this investigation was the same material that is "...used in the the fuel system of the space shuttle to transport fuel from the storage tanks to the combustion section." (1, p. 64)

Because no discussion of the experimental setup could located which was published after the shuttle flight, the expected experiment setup and procedure (as described in a document published prior to the flight) is detailed here: "The screen is placed in between two single sided pieces of copper clad material creating a capacitor. The dielectric of the capacitor consists of the screen and the fluid. In this case freon 113 represents the fuel, which will wick on the screen. The increasing and decreasing of the fluid on the screen will cause the capacitance of the capacitor to change. The changing capacitance will be digitized and stored on two 2k EEPROMs." (1, p. 63)

The experiment was to be initiated at liftoff when noise from the space shuttle main engines activated a series of electronics which were to turn on the experimental setup. Approximately 15 minutes later (at the OMS-1 burn) experimental data documentation was to cease.

Reportedly, at some point during the mission, a Plexiglas case enclosing a seed germination experiment (another of the investigations configured within the can) broke "...spilling a water/formaldehyde mixture inside the GAS can. Several seconds later the batteries and/or controller shorted, ending all experiments except ...[this wicking of fluids experiment] which had its own power supply and controller." (4, p. 34)

Reference (4) reported that because the experiment was equipped with its own power supply and controller, "some results" were obtained. No further details of these results could be located.

Key Words: Capillarity, Capillary Flow, Wicking, Propellant Transfer, Surface Tension, Free Surface, Liquid Transfer, Fluid Management, Liquid/Gas Interface, Solid/Liquid Interface, Electric Field, Rocket Motion, Acceleration Effects, Rocket Noise, Liquid Leakage

Number of Samples: one
Sample Materials: freon 113; fuel cell screening
Container Materials: unknown

Experiment/Material Applications:
It was expected that such a fuel wicking investigation would "...help evaluate the fuel recovery efficiency of the Space Shuttle fuel cells." (1, p. 64)

References/Applicable Publications:
(1) El Paso & Ysleta Schools Get Away Special Payload #34. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, NASA CP-2324, August 1-2, 1984, pp. 59-68. (preflight)

(2) Cargo Systems Manual: Gas Annex for STS 51-G, JSC 17645 51-G, Rev. A, March 20, 1985. (very short description, preflight)

(3) NASA Space Shuttle Mission 51-G Press Kit. June 1985, p. 20. (very short description; preflight)

(4) Ridenoure, R.: GAS mission Summary & Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

(5) G-034 Payload Accommodations Requirements, NASA Goddard Space Flight Center, 1985.

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Capillary Flow Studies

This Consort 1 capillary flow experiment was designed to study the role of wetting in fluid transfer.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (three other experiments, Chapters 1 and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments) the apparatus was also used to (a) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (b) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (a) aligned or (b) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned,

material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted five "Type 2" test wells. Discussion of this specific well-type is detailed here.

Each "Type 2" test well provided the investigator with one experimental opportunity. The well-type used two sample wells, one on the top block and one on the bottom block. Prior to the rocket flight, the well in the upper block of each well-type was filled with water and the well in the lower block of each well-type was filled with triton x-100 (2% in water plus a trace dye). The blocks were then joined together such that the well in the upper block was purposely misaligned with the well on the lower block. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity rocket phase, the upper block again moved to the right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Optical density measurements of each well-pair performed after the rocket flight illustrated the amount of dye which had diffused during the low-gravity phase of the rocket.

Very little discussion of results could be located at this time. However, a short report detailed in Reference (3) appears to couple the results of this experiment with two others by Todd (see also Todd, Consort 1, Phase Rearrangement (Chapter 1); Todd, Consort 1, Turbulent Mixing (Chapter 11)): "Steep surface-tension gradients were established in two types of transport ex-

periments involving two fully-enclosed liquids with an interface between them. When the two liquids were immiscible aqueous solutions [Phase Rearrangement Experiment], no evidence of capillary flow orientation of the phases was obtained during the low-gravity period. When the two liquids were miscible aqueous solutions (one with detergent [Capillary Flow Experiment] and one without [Turbulent Mixing Experiment]) there was also no capillary flow. This last observation could not be made definitely on the ground, where transport was dominated by convection, owing to the similar densities of the two solutions." (3, p. 7)

No further information discussing specific experiment objectives or results could be located at this time.

Key Words: Capillarity, Capillary Flow, Liquid/Liquid Diffusion, Liquid Transfer, Mass Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Aqueous Solutions, Wetting, Miscible Fluids, Buoyancy Effects Diminished, Surface Tension Gradients, Density Distribution, Contamination Source, Liquid Leakage, Contained Fluids

Number of Samples: five
Sample Materials: Top wells: water; bottom wells: triton X-100 2% in water plus a trace dye (the trace dye appears to be trypan blue 4% in water)
Container Materials: inert material

Experiment/Material Applications:
A brief note in Reference (1) indicated that this investigation had commercial applications in the area of bioseparation technology.

No further information discussing the applications of this investigation could be found.

References/Applicable Publications:
(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter on 1/91)

(5) Letter from P. Todd (NIST) to J. M. Cassanto (ITA) dated May 9, 1989 which described experimental analysis to date. (provided by ITA to C. A. Winter, 1/91)

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CHAPTER 3

COMBUSTION STUDIES

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Experiment Origin: USA
Mission: Skylab SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: February 1974 (Month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels, located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Materials Processing Facility (M512): flammability container configured with a specimen holder, water spray nozzle, and photographic equipment
Builder of Processing Facility: NASA Marshall Space Flight Center, Process Engineering Laboratory, Huntsville, Alabama

Experiment:
Zero Gravity Flammability (M479)

The flammability of materials under low-gravity conditions is a significant concern especially when the design of space vehicles is being considered. Although extensive flammability testing of materials has been performed on Earth, combustion processes are significantly different in space. Under low-gravity conditions, there is little or no convective flow to deliver oxidants to the flame or remove combustion products. Such a condition influences most phases of combustion (including smoke toxicity).

The objectives of this SL-4 experiment were to determine (1) the extent of surface flame propagation, (2) the extent of flame flash-over to adjacent materials, (3) the rates of surface and bulk flame propagation, (4) the conditions for self-extinguishment, and (5) the effectiveness of extinguishment by either (a) vacuum or (b) water spray. The experiment was also designed to verify theoretical expectations of flame behavior in a diffusion controlled environment.

Thirty-seven samples of six different materials were employed during the Skylab Experiment: (1) aluminized Mylar film, (2) polyurethane foam, (3) nylon sheet, (4) neoprene-coated nylon fabric, (5) bleached cellulose paper, and (6) Teflon fabric. During the investigation, "Each sample was supported by a metallic frame and ignited by an electrically heated filament. A flammability specimen holder was the mechanical and electrical

interface between the flammability sample and the zero-gravity connector in the work chamber.... The holder positioned the specimen in the approximate center of the chamber, in view of the 16-millimeter data acquisition camera." (13, p. 12-52) The camera used an f-stop of 2.8, a 1/60 shutter speed, an 18 mm lens, and #3443 color infrared film. All samples were ignited and burned in the Skylab atmosphere: 65% oxygen, 35% nitrogen at 5.2 psia. <Note: The "zero-gravity connector" is an electrical connector specially designed to prevent premature electrical activation of a device. (It was especially needed in oxygen-rich environments.)>

Typically, a sample was mounted in the sample holder and then placed inside the chamber onto the zero-gravity connector. After the sample number had been filmed the sample was ignited. The camera ran, automatically, for a preset period of time. The sample was then extinguished by either (1) allowing the sample to burn out by itself (self-extinguishment), (2) opening a vent line to space (vacuum quench), or (3) spraying the sample with water. Six of the samples tested flame flash-over between two strips of material which were separated by gaps of various dimensions.

Post-flight examination of the film and returned samples led to the following conclusions:

(1) Ignition under low-gravity conditions, although not quantitatively evaluated, appeared to be identical in heat and time as on Earth.

(2) Generally, burning rates were much lower in space than on Earth. The low-gravity burning rates were considerably slower than upward or horizontal rates under 1-g conditions.

(3) "Extinguishment by vacuum is effective in that the fire will go out as the available oxygen decreases to some level such as 6 millimeters of oxygen. A significant side effect that deserves consideration is the intensification that develops during the initial phase. The flame can be extremely soft and small with a nearly negligible burning rate and, as the air flow produces a forced convection, it will greatly intensify. If such a procedure was used significantly long the fire could do considerable damage before going out." (2, p. 10)

(4) "Self-extinguishment of an undisturbed fire was not noted." However, "...it can only be said that self-extinguishment is possible but all criteria are undefined." (2, p. 10)

(5) Extinguishment by the application of water is possible if it is controlled and adequate. However, insufficient water striking a burning material results in a momentary flare-up which may (1)

cause burning material to scatter and (2) permit a flash-over resulting in a larger flame. The release of a gaseous extinguishment may prove to be even more detrimental.

(6) Flash-over to adjacent material is possible under low-gravity conditions. However, the upper limit in terms of distance is undefined. Problems with flame visibility made interpretation of this phenomenon difficult.

(7) Experimentation and observation of flammability tests in space is possible. However, the soft, blue flame made filming difficult. The employed photographic setup (f-stop, shutter speed, lens size, and color infrared film) was not adequate.

(8) There did not appear to be a tendency for fuel to become detached and drift as it burned. For samples that melted, cohesive forces drew the unburned material into the fire.

(9) "Large convective forces produced in one-g fires are not characterized in zero-g fires." (2, p. 11)

(10) The visibility of flames was significantly reduced under low-gravity conditions. The fire was luminous if (1) metals were involved or (2) high burning rates were produced.

(11) Fuels that had a large range between melting and boiling temperatures burned with an agitated, pulsating flame that could continue for a significant period of time. The corresponding flame height, on the other hand, may be so small (less than 1/8 inch) as to escape notice and fail to trigger a fire detector.

(Details of each sample may be located in Reference (2))

Key Words: Combustion Studies, Flammability, Burn Rates, Flame Propagation, Flame Extinguishment, Diffusion, Space Vacuum, Water Spray, Quench Process, Convection, Coated Surfaces, Photographic Difficulties

Number of Samples: 37
Sample Materials: aluminized mylar, neoprene-coated nylon fabric, sheet nylon, teflon fabric, bleached cellulose paper, foamed polyurethane
Container Materials: not applicable

Experiment/Material Applications:
The materials selected for this experiment were limited by the number of possible tests and by the number of objectives. The criteria for selection included (1) crew safety, (2) applicability to future spacecraft, (3) stability in an oxygen rich atmosphere over a long period of time, (4) low toxicity of combustion products, and (5) diverse ignition and burning rate flammability situations.

References/Applicable Publications:
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(2) Kimzey, J. H.: Final Report of Skylab Experiment M479 Zero Gravity Flammability. Report Number: JSC-22293, August 1986. (post-flight)

(3) Skylab Zero G Flammability Studies. 16 mm Movie, approximately 30 minutes in length, Movie #JSC 74-652 (post-flight)

(4) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station. August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

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(6) Kimzey, J. H.: Skylab Experiment M-479, Zero Gravity Flammability. Initial Report, Johnson Space Center, 21 pp., March 20, 1974. (post-flight)

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(8) "Experiment M479 - Zero Gravity Flammability." In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-18 - 5-25. (post-flight)

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(10)"M512- Materials Processing Facility," In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-1 - 1-18. (processing facility)

(11) Kimzey, J. H.: Flammability During Weightlessness. NASA TM X-58001, May 1966.

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(13) "Zero Gravity Flammability." In MSFC Skylab Mission Report - Saturn Workshop, October 1974, NASA TM X-64814, pp. 12-52 - 12-54.

(14) "JSC-652 Skylab Zero-G and Flammability Studies." 20-minute Recording of Experiment: (a) 16 mm color film with sound; (b) video. Available at NASA Johnson Space Center, Houston, Texas, Public Affairs Office.

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CHAPTER 4

COMPOSITES WITH GASES

Principal Investigator(s): Deruyttere, A. (1)
Co-Investigator(s): Arkens, O. (2), Smeesters, J. (3), Aernoudt, E. (4)
Affiliation(s): (1-4) Katholieke Universiteit, Leuven, Belgium

Experiment Origin: Belgium
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt. Date: September 1973 (month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels, located forward from the Multiple Docking Apparatus (MDA) area; Skylab manned environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Silver Grids Melted in Space (M565)

Several products which are produced by powder and fiber metallurgy are intentionally porous (e.g., self-lubricating bearings, filters, damping devices). The required cohesion of the material in highly porous products can only be achieved by solid-phase sintering. On Earth, any significant melting results in a collapse of the porous structure. However, if melting is performed in a low-gravity environment, then (1) collapse of the porous structure might be avoided and (2) a product with a unique porous structure might be obtained. Further, certain material properties (e.g., strength, filtering capability) of these low-gravity porous products may be different from those produced on Earth.

"The forces of gravity on a liquid metal are much greater than the forces of cohesion. Consequently, a solid metal structure on Earth loses its shape when melted. In a weightless condition, a liquid metal would be subject to surface tension forces only, which would tend to draw the melt into a spherical shape.

"By using a specific configuration for the original material, or by applying physical constraints to the specimen, other shapes might be achieved." (9, pp. 29-30)

This Skylab, SL-3 experiment was the first in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials. (Deruyttere's other experiments can be found in Chapter 5: "Composites with Solid Particles.") The specific objective of this experiment was to determine the effect of gravity on the

melting of a porous material. This objective was to be achieved by (1) melting and solidifying specimens with specific patterns of pores (in space and on Earth), (2) studying the size, shape, and distribution of the pores after processing, and (3) determining how the surface tension forces reconfigured the patterns in low-gravity and terrestrial environments.

Prior to the experiment, several silver discs containing different pore sizes and shapes were prepared and placed in three silica ampoules designated as Ampoules A, B, and C.

Ampoule A contained eight silver discs (14 mm diameter, 0.1 mm thick). Prior to launch, one or more round, square or hexagonal holes were spark cut or drilled into each of the discs:

Disk 1: 21 round holes of 2 mm diameter and 0.4 mm apart
Disk 2: 14 round holes of 2 mm diameter and 0.8 mm apart
Disk 3: 21 round holes of 1 mm diameter and 1.6 mm apart
Disk 4: 9 round holes of 1 mm diameter and 3.2 mm apart
Disk 5: 4 square holes of 1 mm/side and 6.4 mm apart
Disk 6: 1 central square hole of 1 mm/side
Disk 7: 1 central hexagonal hole of 3.5 mm/side
Disk 8: 1 central hexagonal hole of 3.5 mm/side

Ampoule B contained the same discs as Ampoule A except that the discs were 0.5 mm thick.

"The silver discs were held apart by silica ring spacers and, in order to avoid mixing of the melting products from different discs, the silica ampoules were sealed under a vacuum of about 10^{-5} mm Hg. Before sealing they were heated for one hour at 350 ^{o}C in the same vacuum in order to decompose any Ag_2O present." (2, p. 163)

Ampoule C contained a single sample of silver fibers. Prior to the flight, "The fibres [0.4 mm in diameter, 10-15 mm in length] had been poured as randomly as possible into a cylindrical die, compressed into a disk of about 4 mm high and 50 mm diameter (maximum pressure about 10 kg/mm^2). The disc was sintered in hydrogen for two hours at 900 ^{o}C. From this disc a prism was cut to dimensions 40 x 14 x 4 mm. The porosity was 30% for the Skylab sample and 33% for the ground based test sample." (2, p. 164)

Each ampoule was contained in a stainless steel cartridge. During the mission, the three sample cartridges were melted and solidified in the M 518 Multipurpose Furnace. The cartridges had been made "...to provide a small temperature gradient in the hot zone." (2, p. 161) It was thus anticipated that only some of the samples [discs and single fibre sample] would melt, and those

that would melt "...would stay only a short time at temperatures at or slightly above the melting point." (2, p. 161) Reportedly, thermocouples could not be placed in the sample cartridges. Instead, one was located in the graphite heat leveler (between heater and experiment chamber) and one was located in the heat-extractor plate.

Post-flight examination of the hot thermocouple data indicated that (1) the temperature at this location rose from 35 ^{o}C to 1035 ^{o}C (maximum) in 3 1/2 hours and (2) the temperature was held at at 1035 ^{o}C for one additional hour. <Note: Although not specified, it is assumed that the 'hot' thermocouple refers to that located in the heat leveler.> The furnace power was then turned off and the samples were allowed to cool passively. The initial cooling rate was reported as 38 ^{o}C for the first 5 minutes.

Examination of the flight samples and similarly processed ground-based samples was performed using radiographic, metallographic, and scanning electron microscopic (SEM) techniques (see Reference (1) for detailed discussions of each sample).

<Note: Details of the results from the space- and ground-processed fiber samples can be found in Reference (2). The following summary concentrates on the results attained from examination of the discs in Ampoules A and B.>

Radiography techniques of Ampoules A and B indicated that (1) five of the eight discs had melted in both the Skylab A and B ampoules, while (2) four of eight discs had melted in the ground-based A ampoule and five of eight discs had melted in the ground-based B ampoule.

Further examination indicated that the Skylab samples which did melt were more spherical than the ground-based specimens (except for one sample which contacted a silica ring and wetted it). All of the plates of 0.5 mm diameter became a single drop of molten metal. The 0.1 mm diameter plates either (1) became a single drop or (2) split into several small droplets.

SEM studies of Ampoule B samples indicated that "...even if they had no contact during solidification with... [either]... the stainless steel... [or]... the silica, the spheres would have been far from perfect.... Even if the spheres... [had]... been perfect in the liquid state, on solidification a network of grooves... [had]... appeared on their surface and a shrinkage pipe... [had]... been formed. The grooves... [were]... the traces of a cellular solidification substructure. Cellular solidification has been found in many systems, although observations are generally made on decanted solid-liquid interfaces....

The cell formations... [were]... attributed to constitutional supercooling which itself... [was]... due to impurities which segregate to or from the cell boundaries...." (2, p. 165)

From the examinations of all of the low-gravity samples (including the fiber sample), the following conclusions were reported:

(1) The majority of the original porosity in the samples had disappeared during the melting stage of processing. This behavior was favored due to (a) original open pore structure, (b) the thermal gradient present in the samples, and (c) the low pressure in the ampoules. By adjusting these parameters, a reduction in the loss of porosity could probably be achieved.

(2) The shape and surface condition of the low-gravity samples was determined by (a) surface tension, (b) shrinkage resulting in the formation of a pipe-like shape, and (c) constitutional supercooling caused by impurities which may result in a cellular substructure.

(3) Under low-gravity conditions, impurities at the surface appeared to be slow in leveling out the concentration gradients. Therefore, the action of diffusion and convection appeared to be reduced.

(4) In the partially melted, low-gravity samples, the tendency of the molten portion to become spherical seemed restricted.

Key Words: Composites with Gases, Powder Metallurgy, Fiber Metallurgy, Fibers, Metals, Porosity, Melt and Solidification, Bubble Dispersion, Diffusion, Convection, Segregation, Solutal Gradients, Surface Tension, Surface Energy, Sphericity, Drops, Drop Formation, Wetting, Thermal Gradient, Supercooling, Passive Cooling, Solidification Rate, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Surface Morphology, Cellular Morphology, Material Strength, Filtration, Impurities, Sample Shrinkage, Sample Deformation, Vacuum, Incomplete Sample Processing

Number of Samples: 17
Sample Materials: 16 silver disks into which holes had been drilled or cut (Ampoules A and B) and one single porous sample composed of silver fibers (Ampoule C)
(Ag*)

Container Material: silica ampoules each contained in a stainless steel cartridge
(Si*O*)

Experiment/Material Applications:
This research has applications in the production of components made by powder or fiber metallurgy (e.g., self-lubricating bearings, filters, damping devices). These products require a high porosity where the required material cohesion is obtained by solid-phase sintering.

Silver was selected as the experimental material because it (1) is a typical metal (readily available with high purity), (2) is fairly inexpensive, (3) is easy to roll and draw (for complicated sample shapes), and (4) melted below the maximum available temperature.

References/Applicable Publications:
(1) Deruyttere, A., Aernoudt, E., Goeminne, H., Smeesters, J., Arkens, O., and Verhaeghen, M.: Silver Samples Melted in Skylab Experiment M565. In ESRO Processing and Manufacturing in Space, pp. 27-44. (post-flight) <Note: The publishing date of this document is unclear.>

(2) Deruyttere, A., Aernoudt, E., Goeminne, H., Smeesters, J., Arkens, O., and Verhaeghen M.: Silver Samples Melted in Space, Skylab Experiment M565. In the Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. 1, June 1974, Marshall Space Flight Center, Huntsville, Alabama, pp. 159-203. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low G Measurements at NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(4) "Experiment M565-Silver Grids Melted in Space". In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-80 - 5-82. (post-flight)

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(8) Input received from Principal Investigator, A. Deruyttere, June 1988.

(9) "Silver Grids Melted in Space (M565)." In Skylab Experiments, Information for Teachers, Including Suggestions on Relevance to School Curricula, Vol. 3, Materials Science, NASA, pp. 29-30. (preflight)

(10) Input received from L. Froyen (Katholieke Universiteit), August 1993.

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Co-Investigator(s): Greenwell, E. N. (2)
Affiliation(s): (1) During SPAR 1: Battelle Northwest Laboratories, Richland, Washington, Currently: Cummins Diesel, Columbus, Indiana; (2) Battelle Northwest Laboratories, Richland, Washington

Experiment Origin: USA
Mission: SPAR 1
Launch Date/Expt. Date: December 1975
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Modified Temperature Control Unit (TCU) Furnace
Builder of Processing Facility: Provided by The National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama. Modified by Battelle Northwest Laboratories, Richland, Washington

Experiment:
Feasibility of Producing Closed Cell Metal Foams from Sputter-Deposited Inert Gas Bearing Metals and Alloys (74-10/1)

When metallic foams are produced on Earth, an uneven distribution of trapped gas within the molten metal often occurs. This distribution results because density differences between the liquid and gaseous phases promote separation of the phases. In a low-gravity environment, the buoyancy forces driving the separation are greatly reduced.

This SPAR 1 sounding rocket experiment was the first in a series of experiments designed by Patten et al. to study the feasibility of producing closed-cell metal foams from sputter-deposited, inert gas-bearing metals. The objectives of the experiment centered on the evaluation of "...the effects of gas concentration, melt temperature and time at melt temperature on [1] foam structure, and [2] foaming kinetics." (4, p. 5)

Prior to the rocket flight, the SPAR samples were prepared using high rate sputter deposition techniques. During this ground-based deposition, a controlled quantity of inert argon was trapped in six pure aluminum samples. (Three different gas concentration quantities were investigated (two samples of each concentration).)

During the rocket flight, the samples were rapidly heated within the Temperature Control Unit (TCU) furnace above their melting point of 660 ^{o}C to approximately 1000 ^{o}C. This allowed "...the inert atoms to coalesce, produce bubbles, and expand to provide a

closed cell structure." (5, p. IV-5) The materials were then cooled with a water quench system. "On cooling, the foam solidifies and the atmosphere in each bubble is [a] high purity, low pressure inert gas, effectively a high quality vacuum." (5, p. IV-5)

Post-flight analysis of the six rocket samples were compared to twelve similarly processed terrestrial samples. Metallographic examinations indicated that metal foams were successfully produced in both the rocket and terrestrial experiments. Detailed information about each of the samples was tabulated and included (1) cell size and distribution statistics, (2) trapped argon content, (3) time above melt temperature, and (4) cooling rate through the melting point. Reportedly:

(1) "Detailed analysis of the scatter in cell size, number density and related features... [would] not be attempted since a sufficiently large number of samples was not available to allow reliable determination of trends. However, it seemed generally true that gas content variations up to 250 ppm were not as strongly influential on foaming behavior as expected." (1, p. II-23) <Note: "ppm" was not defined.>

(2) "Trends were observed toward fewer cells/unit volume, less scatter in the number of cells/unit volume, and a larger median cell size in space-processed samples than in ground-processed samples. No trends were observed in mean cell size or scatter in mean cell size." (1, p. II-42)

(3) "...times at which melting occurred on heating for the space processed samples were taken as the beginning of thermal arrests observed in the time-temperature data. Each of these thermal arrests occurred within a few degrees of the published 660^{o}C melting point of aluminum. Times at which solidification occurred were taken as the times when the samples cooled through 660^{o}C. Cooling rates here were very rapid so very little time error was involved with this measurement." (1, p. II-23)

Key Words: Composites with Gases, Metallic Foams, Metals and Alloys, Binary Systems, Melt and Solidification, Solidification Rate, Sputter-Deposition, Closed Cell Foam, Gas Concentration, Bubble Formation, Bubble Dispersion, Liquid/Gas Dispersion, Liquid/Gas Interface, Density Difference, Buoyancy Effects Diminished, Phase Separation, Solid/Liquid Interface, Cellular Morphology, Quench Process, Space Structures, Vacuum

Number of Samples six
Materials: pure metal-inert gas system Al-Ar
(Al*Ar*)
Container Materials: quartz ampoules
(Si*O*)

Experiment/Material Applications:
These experiments were designed to investigate the possibility of producing a new class of metallic foams; foams in which cell size and foam density (for example) could be controlled. Such materials are often useful for structural applications when lightweight, high impact characteristics are important. Other applications of the investigative principles involved in this experiment included hydrogen storage battery systems and reactor fuel research.

The pure metal-inert gas system Al-Ar was chosen because (1) its melting point was well within the operating limits of the TCU furnace, (2) pure Al target material was readily available, (3) published data on argon trapping in aluminum were accessible, and (4) aluminum foam was seen to be of commercial importance.

References/Applicable Publications:
(1) Patten, J. W. and Greenwell, E. N.: Feasibility of Producing Closed-Cell Metal Foams in a Zero Gravity Environment from Sputter-Deposited Inert Gas-Bearing Metals and Alloys. In Space Processing and Applications Rocket Project, SPAR 1 Final Report, NASA TM X-3458, pp. II-1 to II-42, December 1976. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

(3) Patten, J. W. and Greenwell, E. N.: Closed Cell Foams Produced from Sputter Deposited Aluminum--Experiments in Earth and Space. AIAA Paper #77-193, January 1977. (post-flight)

(4) Naumann, R. J. (editor): Feasibility of Producing Closed Cell Metal Foams in a Zero Gravity Environment from Sputter-Deposited, Inert Gas-Bearing Metals and Alloys. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, NASA Technical Memorandum NASA TM-78217, January 1979, pp. 5-6. (post-flight)

(5) Patten, J. W. and Greenwell, E. N.: Feasibility of Producing Closed Cell Metal Foams in a Zero Gravity Environment from Sputter-Deposited Inert-Gas Bearing Metals and Alloys. In Space Processing and Applications Rocket Project, SPAR II, Final Report, NASA TM X-78125, pp. IV-1 - IV-46, November 1977. (post-flight; SPAR 2)

(6) Input received from Experiment Investigator, July 1989.

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Co-Investigator(s): Greenwell, E. N. (2)
Affiliation(s): (1) During SPAR 2: Battelle Northwest Laboratories, Richland, Washington, Currently: Cummins Diesel, Columbus, Indiana; (2) Battelle Northwest Laboratories, Richland, Washington

Experiment Origin: USA
Mission: SPAR 2
Launch Date/Expt. Date: May 1976
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Two Modified Temperature Control Unit (TCU) Furnaces
Builder of Processing Facility: Unknown. However, both The National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama, and Battelle Northwest Laboratories, Richland, Washington, modified the TCUs for this flight.

Experiment:
Feasibility of Producing Closed-Cell Metal Foams from Sputter-Deposited Inert Gas-Bearing Metals and Alloys (74-10/2 and 74-10/3)

This SPAR 2 sounding rocket experiment was the second in a series of experiments designed by Patten et al. to study the feasibility of producing closed cell metal foams from sputter-deposited, inert-gas bearing metals (see Patten, SPAR 1). The specific objectives of the investigation were similar to those outlined in Patten, SPAR 1.

Prior to the rocket flight, the samples were prepared using high rate sputter depositioning techniques. During this ground-based deposition, a controlled quantity of inert argon was trapped in twelve pure aluminum samples. (Three different gas concentration quantities were investigated.)

The twelve samples were placed in two TCU furnaces and processed during the rocket flight. Within the first furnace/sample system (designated as experiment 74-10/2), specimens were heated above the 660 ^{o}C melting point for approximately 85 seconds. Within the second furnace/sample system (designated as experiment 74-10/3), specimens were heated above the 660 ^{o}C melting point for approximately 160 seconds. All samples were then cooled with a water quench system.

Post-flight analysis indicated that nine of the twelve rocket samples were suitable for further, extensive examinations. These samples were compared to similarly processed terrestrial samples. Metallographic examinations indicated that metal foams were produced in both the rocket and terrestrial experiments. Detailed information about each of the samples was tabulated and included (1) trapped argon content, and (2) time above melt temperature. Reportedly:

"Very uniform cell size foams were produced in 1 g in one series of experiments, possibly because a very thick oxide scale was allowed to form, thus, providing uniform constraints to the samples. Bubble coarsening and a larger void volume fraction were observed with increasing time above melting point. In other 1 g experiments and in all zero g experiments, the oxide scales fractured during expansion of the foam, providing non-uniform sample constraint. <Note: It appears that all of the rocket samples had an oxide film.> In the thickest samples foamed in zero g, much more bubble coarsening and a larger void volume fraction were observed with increasing time above the melting point. However, the effects of the oxide scale were still quite pronounced and kinetic information on foam formation behavior was not obtained. It is also believed that much more difference would be noted between ground-based and zero g foam behavior without mechanical restriction from oxide scale." (1, p. IV-3)

Key Words: Composites with Gases, Metallic Foams, Metals and Alloys, Binary Systems, Melt and Solidification, Sputter-Deposition, Closed Cell Foam, Gas Concentration, Bubble Formation, Bubble Dispersion, Coarsening, Liquid/Gas Dispersion, Liquid/Gas Interface, Thin Films, Oxide Layer, Coated Surfaces, Voids, Density Difference, Buoyancy Effects Diminished, Phase Separation, Solid/Liquid Interface, Quench Process, Space Structures

Number of Samples: twelve
Sample Materials: pure metal-inert gas system Al-Ar
(Al*Ar*)
Container Materials: quartz ampoules
(Si*O*)

Experiment/Material Applications:
See Patten, SPAR 1.

References/Applicable Publications:

(1) Patten, J. W. and Greenwell, E. N.: Feasibility of Producing Closed Cell Metal Foams in a Zero Gravity Environment from Sputter-Deposited Inert-Gas Bearing Metals and Alloys. In Space Processing Applications Rocket Project, SPAR II, Final Report, NASA TM X-78125, pp. IV-1 - IV-46, November 1977. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No, NAS5-23438, Mod. 23, ORI, Inc. Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

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(5) Input received from Experiment Investigator, July 1989.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3
Launch Date/Expt. Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Germany

Experiment:
Pores in Al-Alloys

This TEXUS 3 experiment was the first in a series of investigations designed by Hoffmeister et al. to study the low-gravity solidification of a gas/metallic-matrix material. The specific objective of the experiment was to study the interaction between the gas bubbles and solidification front in the molten material.

<Note: The specific TEXUS 3 experimental setup and expected in-flight procedures were not detailed in the available publications. However, Reference (2) implies that they were the same as the TEXUS 3b experiment. The following two paragraphs were written based on Reference (2) and the assumption that the TEXUS 3 and TEXUS 3b setups were similar.>

Prior to flight, three Al-based samples were prepared: (1) Al 99.999 wt.%, (2) Al-2.27 wt.% Mg, and (3) Al-0.67 wt.% Mg. The samples were prepared such that "...the cumulative pore frequency was 50% for a pore diameter of 0.48, 0.34, and 0.28 in sample (1), (2), and (3) respectively." (2, p. 320) All three samples were placed in a single cartridge under an atmosphere of N_2-H_2 (9:1). X-ray pictures were taken of all three samples prior to the rocket flight.

Shortly after the SPAR 3 rocket was launched, the samples were melted and directionally solidified in the TEXUS Experiment Module TEM 01-2. The solidification was induced by the application of He gas to the bottom of the cartridge. The samples were solidified such that the solidification front of the (1) pure aluminum sample was planar, (2) Al-2.27 wt.% Mg sample was cellular, and (3) Al-0.67 wt.% Mg sample was dendritic.

Reportedly, because of a rocket despin failure, TEXUS 3 did not achieve the desired low-gravity level.

It appears that post-flight x-ray pictures were taken of all three samples. It was briefly reported that "...residual accelerations in the order of 0.2 g caused the segregation of the bubbles." (2, p. 320)

Further documentation of the results of this TEXUS 3 experiment does not appear to be available.

The experiment was reflown on TEXUS 3b (see Hoffmeister, TEXUS 3b).

Key Words: Composites with Gases, Metals and Alloys, Metallic Matrix, Binary Systems, Melt and Solidification, Directional Solidification, Porosity, Bubble Formation, Bubble Dispersion, Liquid/Gas Dispersion, Liquid/Gas Interface, Solidification Front Physics, Solid/Liquid Interface, Planar Solidification Interface, Cellular Morphology, Dendritic Solidification, Dendritic Structure, Quench Process, Segregation, Rocket Motion, Acceleration Effects, Rocket Despin Failure

Number of Samples: unclear, possibly three
Sample Materials: aluminum alloys
(Al*)
Container Materials: unknown

Experiment/Material Applications:
See Hoffmeister, TEXUS 3b.

References/Applicable Publications:
(1) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

(2) Pores in Aluminum Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 320-321. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3b
Launch Date/Expt. Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Germany

Experiment:
Pores in Al-Alloys

This TEXUS 3b experiment was the second in a series of investigations designed by Hoffmeister et al. to study the low-gravity solidification of a gas/metallic-matrix material (see Hoffmeister, TEXUS 3). The specific objective of the experiment was to study the interaction between the gas bubbles and solidification front in the molten material.

Prior to flight, three Al-based samples were prepared: (1) 99.999 wt.% Al, (2) Al-2.27 wt.% Mg, and (3) Al-0.67 wt.% Mg. The samples were prepared such that "...the cumulative pore frequency was 50% for a pore diameter of 0.48, 0.34, and 0.28 in sample (1), (2), and (3) respectively." (1, p. 320) All three samples were placed in a single cartridge under an atmosphere of N_2-H_2 (9:1). X-ray pictures were taken of all three samples prior to the rocket flight.

Once the SPAR 3b rocket had been launched and low-gravity conditions achieved, the samples were melted and directionally solidified in the TEXUS Experiment Module TEM 01-2. The solidification was induced by the application of He gas to the bottom of the cartridge. The samples were solidified such that the solidification front of the (1) pure aluminum sample was planar, (2) Al-2.27 wt.% Mg sample was cellular, and (3) Al-0.67 wt.% Mg sample was dendritic.

Post-flight x-ray pictures were taken of all three samples. It was reported that the pores had accumulated in the upper portion of the samples indicating the material was solidified from the bottom and along the axis. The gas pore size also increased and the total number of pores decreased during processing. The pore diameters had increased by factors of 1.32, 1.27, and 1.25 for samples (1), (2), and (3), respectively.

"It was concluded that the bubbles coagulated in the melt. It was assumed that the gas pressure was a maximum just before the beginning of the solidification and that the increase of the total pore volume between the bottom and the top of the samples was induced by the axial thermal gradient. Accordingly, the largest expansion was observed in sample (3), located at the top of the cartridge, and the second largest in sample (1) located in the middle." (1, p. 320) <Note: It is not clear what is meant by "expansion" since the largest increase in pore diameter occurred in sample (1).>

Key Words: Composites with Gases, Metals and Alloys, Metallic Matrix, Metallic Foams, Binary Systems, Melt and Solidification, Directional Solidification, Pores, Porosity, Pore Size, Bubble Formation, Coagulation, Bubble Growth, Bubble Dispersion, Liquid/Gas Dispersion, Liquid/Gas Interface, Solidification Front Physics, Solid/Liquid Interface, Thermal Gradient, Planar Solidification Interface, Cellular Morphology, Dendritic Solidification, Dendritic Structure, Quench Process, Gas Pressure, Arc Welding

Number of Samples: three
Sample Materials: (1) 99.999 wt.% Al, (2) Al-2.27 wt.% Mg, and (3) Al-0.67 wt.% Mg
(Al*, Al*Mg*)
Container Materials: unknown

Experiment/Material Applications:
This research is related to the study of (1) arc welding and (2) the production of metallic foams.

The specific reasons why Al and Al-Mg alloys were selected for this experiment were not detailed in the available publications.

References/Applicable Publications:
(1) Pores in Aluminum Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 320-321. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: SPAR 9
Launch Date/Expt. Date: January 1981
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: General Purpose Rocket Furnace (GPRF): One of the three available heated cavities in the GPRF was dedicated to this experiment.
Builder of Processing Facility: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Foam Copper (77-9)

This SPAR 9 experiment was the first in a series of investigations designed by Pond et al. to study the formation of copper foam under low-gravity conditions.

The specific experimental objectives and setup were not detailed in the available publication (listed below), but were probably similar to those outlined under a similar experiment performed by Pond et al. on SPAR 10 (see Pond, SPAR 10).

During the SPAR 9 mission, the sample cartridge, which was held in module 1 of the General Purpose Rocket Furnace (GPRF), reportedly "...failed to reach processing temperature (1150 ^{o}C) because the internal potentiometer settings that determine the temperature profile were never verified, due to the concern of over-stressing the heater windings by extra [ground-based] testing." (1, p. II-7) During the flight, the module temperature reached a maximum of 1070 ^{o}C.

Post-flight examination of the module indicated that the potentiometer settings were correct for 1070 ^{o}C. "Hence, the GPRF performed exactly as it was programmed, but the potentiometer settings were in error." (1, p. II-11) The experiment was reflown on SPAR 10 (see Pond, SPAR 10).

No other information concerning this experiment could be located at this time.

Key Words: Composites with Gases, Melt and Solidification, Metallic Foams, Bubble Formation, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Incomplete Sample Processing

Number of Samples: one
Sample Materials: copper
(Cu*)
Container Materials: unknown

Experiment/Material Applications:
Please refer to Pond, SPAR 10.

References/Applicable Publications:
(1) Poorman, R. (Compiler): SPAR IX Post Flight Engineering Report. In Space Processing Applications Rocket (SPAR) Project, SPAR IX Final Report, NASA TM-82549, January 1984, pp. II-1 - II -19 (specifically, p. II-7). (post-flight)

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Experiment Origin: USA
Mission: SPAR 10
Launch Date/Expt. Date: June 1983
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Low-Gravity Exothermic Heating/Cooling Apparatus
Builder of Processing Facility: Unknown

Experiment:
Foam Copper (77-9)

This SPAR 10 experiment was the second in a series of investigations designed by Pond et al. to study the formation of a copper foam under low-gravity conditions (see Pond, SPAR 9). The specific objective of the experiment was to produce a foam copper-copper oxide alloy with (1) a homogeneous porosity and (2) a density of less than one-third that of pure copper. The experiment was to use the following reaction to produce bubbles of CO within the copper melt:

$Cu_2O(1) + C$ (graphite) --> $2\ Cu(1) + CO$

(This reaction has been used for many years in the fire refining of copper.) "The advantage here is that the process is not limited by the solubility of a particular gas in a molten or solid phase. The amount of gas which can be generated is limited only by the reactants, and indeed,... limiting the reactants is the way to control the amount of gas generated." (1, p. 76) (Reference (1) contains a discussion of the fundamental considerations, thermodynamics, and extensive ground-based studies performed to investigate the rate and quantity of gas evolved during the reaction.)

Prior to the SPAR 10 mission, the sample was prepared by mixing six grams of copper powder with 60 mg of carbon and 0.47 g of Cu_2O powders (see Reference (1) for further sample preparation procedures). The sample was configured in a TZM molybdenum alloy cartridge (0.812 inch outer diameter; 2.75 inches long; 0.15 inch wall thickness). The cartridge contained a helical spring to prevent sample movement during handling and rocket launch. The cartridge was sealed by TIG-welding to maintain the expected

pressure development.

During the SPAR 10 experiment, the Low Gravity Exothermic Heating/Cooling Apparatus was used to melt and resolidify the sample. Reportedly, it was clear that the expected reaction between the carbon and copper oxide occurred. Yet, "...the temperature measurement of the capsule was lost; and therefore, it is not known whether or not the sample solidified before it became affected by the Earth's gravitation. However, the reclaimed capsule provided evidence that a copper foam was developed although it was not maintained.

"In the reclaimed capsule, the copper was coated onto three-quarters of the inner surface of the cavity in the half nearest the TIG-welded end. This copper movement could have been the result of... [the copper]... wetting the surface of the TZM or that... [the copper]... developed as a foam which collapsed onto the surface. Isolated in this surface coating are 17 crystals of copper approximately 2 mm on an edge and extending from the surface 2 to 3 mm. This indicates that the pool of copper from which these crystals grew must have been at least 2 to 3 mm thick. Such crystals could not have been developed from a copper sheet generated by wetting but could have developed from a foam. Although this evidence strongly suggests the development of a copper foam, there is no evidence to support a reason for its collapse since the experiment may have been subject to relatively high 'g' forces before solidification." (1, p. 86) <Note: It was reported that further investigation of this phenomenon is expected and will be reported at a later date.>

Key Words: Composites with Gases, Metallic Foams, Foam Stability, Metallic Matrix, Metals and Alloys, Binary Systems, Reactant Solutions, Melt and Solidification, Porosity, Bubble Formation, Bubble Dispersion, Gas Formation, Liquid/Gas Dispersion, Homogeneous Dispersion, Segregation, Liquid/Gas Interface, Solid/Liquid Interface, Powders, Crystal Morphology, Reaction Kinetics, Wetting, Wetting of Container, Density Decrease, Coated Surfaces, Acceleration Effects, Processing Difficulties

Number of Samples: one
Sample Materials: Cu-Cu_2O-C
(Cu*O*C*)
Container Materials: TZM (Mo-0.5Ti-0.08Zr-0.015C)
(Mo*Ti*Zr*C*)

Experiment/Material Applications:
Metallic foams have many possible applications where light, strong materials are required (e.g., structural members in aircraft and automobiles). Metal foams are created by the formation of bubbles within a melt. On Earth, because of gravitational effects (buoyancy), the bubbles segregate and a homogeneous dispersion cannot be obtained.

References/Applicable Publications:
(1) Pond, R. B., Sr. and Winter, J. M., Jr.: SPAR Experiment 77-9/1R SPAR X Foam Copper. In Space Processing Applications Rocket (SPAR) Project, SPAR X Final Report, NASA TM 86548, pp. 75-86, July 1986. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 4
Launch Date/Expt. Date: May 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Four-Chamber Isothermal Heating Facility Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Steel Foams (Part 1 of 2)

This TEXUS 4 experiment was the fifth in a series of investigations designed by Pötschke and/or Neuschütz et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b (Chapter 5)). The emphasis of the investigation was to examine composites with solid (Al_2O_3) and gaseous inclusions.

The experiment was also the first of a two-part investigation designed by Pötschke et al. and performed during the TEXUS 4 mission.

The summary of the objectives, experimental setup, and results from both parts of the experiment are found in Pötschke, TEXUS 4 (this chapter), Metallic Steel Foams (Part 2 of 2).

Key Words: Composites with Gases, Composites with Solid Particles, Metals and Alloys, Metallic Foams, Metallic Matrix, Binary Systems, Ternary Systems, Multiphase Media, Two-Phase System, Phase Stability, Foam Stability, Melt and Solidification, Bubble Dispersion, Particle Dispersion, Inclusions, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Particle Wetting, Surface Tension, Capillary Forces, Marangoni Convection, Buoyancy Effects, Thermal Soak

Number of Samples: See Pötschke, TEXUS 4, Metallic Steel Foams (Part 2 of 2).
Sample Materials: See Pötschke, TEXUS 4, Metallic Steel Foams (Part 2 of 2).
Container Materials: See Pötschke, TEXUS 4, Metallic Steel Foams (Part 2 of 2).

Experiment/Material Applications:
See Pötschke, TEXUS 4, Metallic Steel Foams (Part 2 of 2).

References/Applicable Publications:
(1) Input received from Principal Investigator, J. Pötschke, September 1989 and August 1993.

(2) Manufacture of Steel Foam. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 322-323. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 4
Launch Date/Expt. Date: May 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Four-Chamber Isothermal Heating Facility Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Steel Foams (Part 2 of 2)

Research has illustrated that liquid steel has no surface elasticity. As a result, spherical foams are only stable for less than 1 second. It is unclear if this short life is due to (1) the buoyancy of the foaming gas bubbles or (2) the capillary forces acting in the system. (The capillary forces are enhanced by the material's high surface tension and temperature dependence (Marangoni convection).)

This TEXUS 4 experiment was the sixth in a series of investigations designed by Pötschke and/or Neuschütz et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b (Chapter 5); Pötschke, TEXUS 4 (Metallic Steel Foams (Part 1 of 2)). The emphasis of the investigation was to (1) examine composites with gaseous inclusions and (2) determine the effect of capillary forces on the stability of steel foams.

<Note: The experiment was also the second of a two-part investigation designed by Pötschke et al. and performed during the TEXUS 4 mission (see Pötschke, TEXUS 4 (this chapter), Metallic Steel Foams (Part 1 of 2). The goals, experimental setup, and results from both parts of the experiment are detailed below.>

The flight experiment was performed in two chambers of the TEM 01 four-chamber isothermal furnace. During the low-gravity phase of the mission, four steel samples (supersaturated with nitrogen) were (1) melted, (2) soaked, and (3) solidified. (Two of the samples were soaked for 120 seconds and the other two were soaked for 240 seconds.)

Post-flight examination of the low-gravity processed samples revealed no gas bubbles within the material. It was reported that this result indicated that a two-phase steel foam is as unstable under reduced gravity conditions as it is on Earth.

No further discussion of the objectives, experimental setup, or results could be located in the available publications.

Key Words: Composites with Gases, Composites with Solid Particles, Metals and Alloys, Metallic Foams, Metallic Matrix, Binary Systems, Two-Phase System, Foam Stability, Phase Stability, Melt and Solidification, Supersaturation, Bubble Dispersion, Inclusions, Sphericity, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Surface Tension, Capillary Forces, Marangoni Convection, Thermocapillary Convection, Buoyancy Effects, Thermal Soak

Number of Samples: four
Sample Materials: steel samples (high speed steel) supersaturated with nitrogen (approximately 0.1% N) <Note: It appears that at least two of the samples (those from Part 1 of the experiment) also contained Al_2O_3 inclusions.>
(Fe*N*)
Container Materials: Alumina
(Al*O*)

Experiment/Material Applications:
Steel foam produced directly from the melt could find applications as a high strength, light-weight material (e.g., for sound proofing or for energy absorbent components).

References/Applicable Publications:
(1) Input received from Principal Investigator, J. Pötschke, September 1989 and August 1993.

(2) Manufacture of Steel Foam. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 322-323. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 6
Launch Date/Expt. Date: May 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2 isothermal furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Steel Foams

This TEXUS 6 experiment was the eighth in a series of investigations designed by Pötschke and/or Neuschütz et al. to study the low-gravity solidification of metallic composites (see Neuschütz TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5 (Chapter 5); Pötschke, TEXUS 4 (two experiments, this chapter). The emphasis of the investigation was to examine composites with gaseous inclusions.

Results from the earlier TEXUS 4 experiment in this investigative series indicated that a two-phase, steel foam was as unstable under reduced gravity conditions as it was on Earth. Therefore, the specific objective of the experiment was to prevent the coalescence of gaseous inclusions in the metallic material (and thus increase foam stability) by including a third-phase material (a graphite lamellar network) in the foaming substance.

Prior to the TEXUS 6 flight, two powder samples (10 mm diameter, 8 mm long) were prepared. Sample 1 was comprised of Fe-4.8% C-1.53% SiO_2 (wt.%) and Sample 2 was comprised of Fe - 4.8% C - 0.11% Al - 1.53% SiO_2 (wt.%). The SiO_2 particles were spherical and had a diameter of 100 microns. Each sample was placed in its own Al_2O_3 crucible. The crucibles were then stacked in a single TZM cartridge.

The experiment was performed in the Texus Experiment Module TEM 01-2 isothermal furnace. During the low-gravity phase of the mission, the samples were heated under ambient atmospheric conditions and quenched with helium. It was intended that CO gas bubbles would form in the samples according to the reaction:
$SiO_2 + 2\ C \rightarrow Si + 2\ CO$

Post-flight examination of Sample 1 revealed that the material had only partially melted. The sample exhibited a foaming ratio of 5% with a density decrease from an initial value of 7.21 g/cm^3 to 6.86 g/cm^3.

Post-flight examination of Sample 2 revealed that the material was only completely melted for 10 seconds. The sample exhibited a foaming ratio of 12% with a density decrease from an initial value of 7.21 g/cm^3 to 6.35 g/cm^3.

It was concluded that the samples were not molten long enough to form "real foams." "Only the initial stages of bubble nucleation and graphite lamellae growth were reached.... It was shown that the graphite lamellae could grow independent from the gas bubbles but that their nucleation was easier at the surface of the bubbles. As the gas bubbles adhered to the graphite due to a bad wetting of the liquid, their coalescence was prevented. Therefore, it could be expected to obtain foams more stable than without graphite." (2, p. 324)

Key Words: Composites with Gases, Metallic Foams, Metallic Matrix, Ternary Systems, Multiphase Media, Phase Stability, Foam Stability, Powder Metallurgy, Melt and Solidification, Bubble Formation, Bubble Nucleation, Bubble Coalescence, Bubble Dispersion, Particle Dispersion, Gas Formation, Inclusions, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Reaction Kinetics, Particle Wetting, Surface Tension, Capillary Forces, Quench Process, Density Decrease, Lamellar Structure, Incomplete Sample Processing

Number of Samples: two
Sample Materials: (1) Fe - 4.8% C - 1.53% SiO_2 (wt.%) and
(2) Fe - 4.8% C - 0.11% Al - 1.53% SiO_2 (wt.%)
(Fe*C*Si*O*, Fe*C*Al*Si*O*)
Container Materials: alumina crucible (Al_2O_3) configured within a TZM cartridge. (TZM is a high temperature resistant molybdenum alloy with small amounts of titanium and zirconium.)
(Al*O*, Mo*Ti*Zr*)

Experiment/Material Applications:
See Pötschke, TEXUS 4.

References/Applicable Publications:

(1) Input received from Principal Investigator, J. Pötschke, September 1989 and August 1993.

(2) Manufacture of Steel Foam. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 324-325. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2 isothermal furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Manufacture of Iron Foam

This TEXUS 7 experiment was the ninth in a series of investigations designed by Pötschke and/or Neuschütz et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5 (Chapter 5); Pötschke, TEXUS 4 (two experiments), TEXUS 6 (this chapter)). The emphasis of the investigation was to examine composites with gaseous inclusions. The specific objective of the TEXUS 7 experiment was to determine if a solid network within a foaming material could prevent the coalescence of gas bubbles.

During the mission, two samples were processed in the TEXUS Experiment Module TEM 01-2. Sample #1 was comprised of Fe - 4.3% P - 0.13% N_2 (wt.%) and Sample #2 was comprised of Fe - 4.8% C - 1.53% SiO_2 (wt.%). (Sample #2 had the same composition as one of the samples flown during the TEXUS 6 mission (see Pötschke, TEXUS 6).)

Post-flight, it was reported that Sample #1 exhibited a foaming efficiency of 44%. This value equaled the foaming efficiency of this material when processed on Earth.

Examination of Sample #2 revealed a foam consisting of a fine network of iron which was interlaced by graphite. Reportedly, the low-gravity sample was very brittle. In a similarly processed, ground-based sample, "...the melt was sucked out of the graphite skeleton due to gravity." (2, p. 326)

"It was concluded that foams consisting of three phases on melting are more stable than those consisting of two phases. However, the size and distribution of the bubbles cannot be con-

trolled using reactive gases, even in microgravity. This could however be possible with inert gases." (2, p. 326)

<Note: Reference (1) contains a summary of the conclusions from the TEXUS 4, TEXUS 5, TEXUS 6, and TEXUS 7 experiments in this investigative series. This reference also includes a comparison of these results with those from other experiments concerning the Cu-C-O system.>

Key Words: Composites with Gases, Metallic Foams, Metallic Matrix, Ternary Systems, Multiphase Media, Phase Stability, Foam Stability, Melt and Solidification, Bubble Formation, Bubble Coalescence, Bubble Dispersion, Bubble Distribution, Particle Dispersion, Gas Formation, Inclusions, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Reaction Kinetics, Particle Wetting, Surface Tension, Capillary Forces, Density Decrease

Number of Samples: two
Sample Materials: (1) Fe - 4.8% C - 1.53% SiO_2 (wt.%) and (2) Fe - 4.3% P - 0.13% N_2 (wt.%)
(Fe*C*Si*O*, Fe*P*N*)
Container Materials: alumina, Al_2O_3
(Al*O*)

Experiment/Material Applications:
See Pötschke, TEXUS 4.

References/Applicable Publications:
(1) Input received from Principal Investigator, J. Pötschke, September 1989 and August 1993.

(2) Manufacture of Iron Foam. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 326-327. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 5
Launch Date/Expt Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: cartridge furnace
Builder of Processing Facility: ERNO Raumfahrttechnik, Bremen, Germany <Note: now called Messerschmitt-Boelkow-Blohm (MBB/ERNO)>

Experiment:
Metallic Foams

This TEXUS 5 experiment was the first in a series of investigations designed by Schäfer to study the production of metallic foams under low-gravity conditions. The specific objective of the experiment was to determine if metallic foams with high porosity could be formed if gravity-driven forces (such as buoyancy) were absent.

Prior to the mission, a sample consisting of 3 g of $CuSn_6$ with additions of CuO and C was placed within an Al_2O_3 crucible. During the low-gravity phase of the rocket flight, the specimen was processed in the TEXUS Experimental Module TEM 01-1 cartridge furnace. During this processing, the material was heated to approximately 1090 ^{o}C (this temperature is above the liquidus temperature) where the following reaction occurred:

$$CuO + C = Cu + CO$$

Before the end of the low-gravity period, the sample was rapidly cooled below the solidus temperature.

Reference samples were prepared under 1-g conditions using the same time/temperature profile.

Post-flight, the 1-g and low-gravity processed samples were compared. It was reported that the 1-g sample contained pores with a maximum diameter of 0.05 mm and had a porosity of 0.8%. The space processed sample contained pores with a maximum diameter of 2 mm and had a porosity of 11%. It was concluded that the stability of a metallic foam is increased under low-gravity conditions.

No further discussion of the results or conclusions could be located.

Key Words: Composites with Gases, Metallic Foams, Metallic Matrix, Multiphase Media, Phase Stability, Foam Stability, Melt and Solidification, Bubble Formation, Bubble Dispersion, Gas Formation, Porosity, Pore Size, Buoyancy Effects, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Reaction Kinetics

Number of Samples: one
Sample Materials: $CuSn_6$ alloy with additions of copper oxide (CuO) and carbon
(Cu*Sn*Cu*O*C*)
Container Materials: Alumina, Al_2O_3 in stainless steel
(Al*O*)

Experiment/Material Applications:
Materials processing under low-gravity conditions increases the stability of gas bubbles in a molten metal. This increased stability is attributed to the absence of gravity-induced buoyancy.

The specific reason why the $CuSn_6$ material was chosen for this experiment was not detailed in available publications.

References/Applicable Publications:
(1) Schäfer, W.: Gasblasen in Metallschmelzen. In Deutsche Gesellschaft für Luft- und Raumfahrt e.V. Spacelab-Nutzung, Werkstofforschung und Verfahrenstechnik im Welraum, Status Seminar 1982 des Bundesministeriums für Forschung und Technologie, pp. 169-176.

(2) Schäfer, W.: TEXUS 5 Experiment: Metallschaumherstellungen. Final Report, Dornier System, 1982.

(3) Input received from Principal Investigator, W. Schäfer, August 1989 and August 1993.

(4) Metallic Foams. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 328-329. (postflight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 9
Launch Date/Expt Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal cartridge furnace
Builder of Processing Facility: ERNO Raumfahrttechnik, Bremen, Federal Republic of Germany <Note: now called Messerschmitt-Boelkow-Blohm (MBB/ERNO)>

Experiment:
Preparation of Aluminum Foams

This TEXUS 9 experiment was the second in a series of investigations designed by Schäfer to study the production of metallic foams under low-gravity conditions (see Schäfer, TEXUS 5). The specific objective of the experiment was to investigate the stability of aluminum foams containing a solid lamellar structure.

Prior to the mission, two aluminum samples were prepared. One of these samples had been pre-processed on Earth and contained gas bubbles; the other had additions of TiH_2. <Note: A more detailed discussion of the pre-processed Earth sample was not presented.> Both samples were placed within alumina crucibles.

During the low-gravity phase of the sounding rocket flight, the materials were melted and resolidified in the TEXUS Experimental Module TEM 01-1 cartridge furnace. During this processing, hydrogen gas bubbles were produced in the sample containing additions of TiH_2 by the reaction:

$$3Al + TiH_2 = Al_3Ti + H_2$$

(It was intended that the Al_3Ti created by the above reaction would form a solid lamellar structure which would then prevent coalescence of the bubbles.)

Similar samples were processed under the same thermal conditions on Earth.

Post-flight, it was reported that the rocket and 1-g processed samples which contained TiH_2 additions had about the same porosity (61% and 67%, respectively). However, the porosity of

the low-gravity sample without the TiH_2 addition increased by a factor of 1.5 over a similar, Earth-processed sample (from 48% to 74%).

No further discussion of the results or conclusions could be located.

Key Words: Composites with Gases, Metallic Foams, Metallic Matrix, Multiphase Media, Phase Stability, Foam Stability, Melt and Solidification, Bubble Formation, Bubble Dispersion, Bubble Coalescence, Gas Formation, Porosity, Pore Size, Buoyancy Effects, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Reaction Kinetics, Lamellar Structure

Number of Samples: two
Sample Materials: Sample 1: aluminum with titanium hydride additions (TiH_2); sample 2: aluminum containing pores
(Al*Ti*H*, Al*)
Container Materials: alumina crucibles (Al_2O_3, in a stainless steel cartridge)
(Al*O*)

Experiment/Material Applications:
See Schäfer, TEXUS 5.

The specific reasons why the aluminum samples were chosen for this experiment were not detailed in the available publications.

References/Applicable Publications:
(1) Schäfer, W.: TEXUS 9 Experiment-Aluminiumschaumherstellung. Final Report, Dornier System, 1984.

(2) Input received from Principal Investigator, W. Schäfer, August 1989 and August 1993.

(3) Preparation of Aluminum Foams. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 330-331. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #6, STS-006 (STS 31-B, Challenger)
Launch Date/Expt. Date: April 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-049
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-049: The United States Air Force Academy, Colorado Springs, Colorado
Processing Facility: A sealed evacuated quartz glass tube, wrapped with nichrome wire and coated in asbestos cement.
Builder of Processing Facility: Principal Investigator, R. R. Neel, and a glass blower at the United States Air Force Academy, Chemistry Department, Colorado Springs, Colorado

Experiment:
Formation of Foam Metal

During the foaming of molten metals on Earth, gaseous bubbles float to the surface of the liquid, preventing an even distribution of foam within the sample. Thus, the specific objective of this STS-006 experiment was to produce a more uniformly dispersed metallic lead foam in the low-gravity environment.

The experiment was one of six investigations housed within the G-049 Get Away Special Canister on STS-006. (Four other experiments (of the six) were applicable to this database (see Amidon, STS-006 (Chapter 14); Gross, STS-006 (Chapter 18); Peter, STS-006 (Chapter 18); Streb, STS-006 (Chapter 14)).)

Prior to the shuttle flight, a 7.5 cm quartz tube containing an 80:1 ratio of lead to sodium bicarbonate was wound with a nichrome wire and configured within the GAS canister. During the low-gravity experiment, the nichrome was heated by passing a 2 amp current (at 16 volts) through the wire. Melting of the lead-based sample, and subsequent foaming of the molten material, was expected. <Note: Reference (2), which was published before STS-006 was launched, indicated that the crucible was to be quenched (via the release of freon-11) after 5 minutes of heating. Post-flight references did not discuss such a quenching procedure and

none of the references further detailed how this freon release was to be accomplished.>

Post-flight, the low-g sample was compared with similarly processed 1-g reference samples. While it was reported that the low-gravity sample did melt and the bicarbonate did outgas, the lead exhibited no signs of foaming. Similarly, the 1-g reference samples exhibited little or no bubbling characteristics. It was hypothesized that "...the surface tension forced the gases out during the cooling phase and the foam could not solidify." (1, p. 318)

No further discussion of this hypothesis was presented and further details concerning the experiment setup or results could not be located.

Key Words: Composites with Gases, Metallic Foams, Metallic Matrix, Two-Phase System, Melt and Solidification, Bubble Formation, Bubble Dispersion, Outgassing, Gas Formation, Homogeneous Dispersion, Buoyancy Effects, Liquid/Gas Dispersion, Liquid/Gas Interface, Surface Tension, Marangoni Movement (Migration) of Bubbles, Solid/Liquid Interface, Quench Process, Space Structures

Number of Samples: one
Sample Material: lead; foaming agent: sodium bicarbonate (Pb*, Na*C*)
Container Materials: quartz (Na*C*)

Experiment/Material Applications:
Building materials constructed from foamed molten metals could have (1) a high strength-to-weight ratio and (2) a density of one-tenth or less that of the original metal. Such materials may be useful for space deployed structures.

References/Applicable Publications:
(1) Worsowicz, Captain C. and Swan, Major P.: The Eaglets have Flown. Space Education, Vol. 1, May 1984, pp. 317-319. (post-flight)

(2) Cargo Systems Manual: GAS Annex for STS-6. December 3, 1982, JSC-17645, Annex STS-6. (very short description; preflight)

(3) STS-6 Getaway Specials, NASA News, NASA GSFC, November 24, 1982. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

(5) NASA STS-6 Sixth Space Shuttle Mission Press Kit, April 1983, pp. 41-43. (preflight)

(6) Input received from Principal Investigator R. R. Neel, June 1991.

(7) Neel, R. R., II: The Get Away Special Foam Metal Experiment. 1983, 16 pp. (preflight)

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Experiment Origin: USA
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facilities, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Bubble Reinforced Materials (1ES311 A&B)

Composite materials generally consist of a dispersed phase (solid or gas) within a matrix. The properties of the matrix material (e.g., strength) are determined not only by the materials themselves, but also the distribution of the second phase through the matrix. The distribution of the second phase is affected by such parameters as (1) surface properties of the dispersed phase, (2) buoyancy forces present while the matrix material is molten, (3) gravity-independent convective flows, and (4) interaction of the dispersed phase with a moving solidification front.

This Spacelab 1 experiment was designed to study the behavior of dispersed phases during a melting and solidification treatment. The specific objectives of the experiment were to (1) investigate the performance of thin film oxide containers, (2) study the behavior of dispersed solid and gaseous phases within a liquid melt, and (3) determine the interaction of the dispersed phases, eutectic structures, and cellular structures with a solidification front.

Prior to the Spacelab flight, a total of 21 samples which contained Ag, Al, or Cu as the base metal were prepared. The samples were either (1) binary alloys (Ag-Cu, eutectic composition), (2) Ag-Cu binary alloys with additions of 0.1% Al_2O_3 particles, (3) Al with fine dispersions of Al_2O_3 (4 and 7 wt.% concentrations), (4) Avional 2024 alloy, or (5) Al with 1% Ni. The Al-Al_2O_3 samples were prepared by hot extrusion and referred to as S.A.P. Samples were prepared using various methods including (1) sintering, (2) compacting only, (3) explosion sintering, (4) alternated electrolytic deposition, (5) hot extrusion, or (6) preparation from slices. The sample shapes included (1) cylindrical with hemispherical extremes, (2) cylindri-

cal with flat extremes, and (3) slices (approximately 1 mm thick). Some of the samples also had one of two coatings: (1) a thin (1000 to 10000 angstroms) alumina coating, or (2) a Ni film coating. Other samples had no coating. All of the samples were placed witnin outgassed graphite cylinders which were configured with thermocouples. (There was approximately a 0.1 mm spacing between the samples and graphite walls.) It appears that (1) the graphite cylinders were encased within Ta cartridges and (2) the cartridges contained Ar gas such that the pressure would reach 1 bar at the melting temperature. <Note: See Reference (1) for a listing of the sample compositions, preparations, and coatings.>

During the Spacelab 1 mission, the Isothermal Heating Facility (IHF) was used to melt and solidify the samples. Corresponding reference samples were processed on Earth for comparison.

Post-flight examination of the low-gravity processed samples led to the following conclusions:

(1) The oxide films maintained the initial sample shape after melting and solidifying for (a) the Ag-Cu materials and (b) the Al samples with fine dispersions of Al_2O_3 particles which had been prepared by hot extrusion.
(2) Only the oxide films on some of the S.A.P. alloys were able to withstand volume expansion.
(3) It appeared that the bubbles within the samples were swept to phase and grain boundaries by the solidification front.
(4) The eutectics formed in low gravity were of a larger lamella spacing than the reference samples. This result probably depended upon smaller solidification rates as well as larger diffusion coefficients and/or smaller nucleation rates. It was also noted that grain sizes and coherency lengths were larger in the low-gravity samples.
(5) The eutectics in the Cu-Ag samples with Ni coating were globular. Microanalytical mapping demonstrated larger Ni diffusion lengths for the low-gravity samples.
(6) After low-gravity processing, "...S.A.P. alloys present specific phenomena of reduced coalescence, leading to a uniform distribution of aggregates of limited size, much smaller than the one of the aggregates forming in 1-g and probably depending on the Brownian motion. After MST [melting and solidification treatment] in 0-g particular cell structures result, with spacings comparable to those between the small aggregates." (1, p. 107)
(7) No evidence of convective motions caused by surface tension gradients was found in the oxide-film coated metals.

Key Words: Composites with Gases, Composites with Solid Particles, Metallic Matrix, Metals and Alloys, Eutectics, Binary Systems, Ternary Systems, Two-Phase System, Multiphase Media, Melt and Solidification, Homogeneous Dispersion, Bubble Dispersion, Bubble Motion, Nucleation, Particle Dispersion, Particle Coalescence, Particle Aggregation, Coated Surfaces, Skin Technology, Thin Films, Oxide Layer, Solid/Liquid/Gas Dispersion, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Buoyancy Effects, Diffusion, Diffusion Coefficient, Brownian Motion, Surface Tension, Surface Tension Gradients, Surface Tension-Driven Convection, Solidification Front Physics, Solidification Rate, Lamellar Structure, Grain Structure, Grain Size, Grain Boundaries, Cellular Morphology, Material Strength, Volume Expansion, Contained Fluids

Number of Samples: twenty-one
Sample Materials: (1) binary alloys (Ag-Cu, eutectic composition), (2) Ag-Cu binary alloys with additions of 0.1% Al_2O_3 particles, (3) Al with fine dispersions of Al_2O_3 (4 and 7 wt.% concentrations), (4) Avional 2024 alloy, and (5) Al with 1% Ni. (See Reference (1) for details.)
(Ag*Cu*, Ag*Cu*Al*O*, Al*Ni*)
Container Materials: graphite sample holder (see Reference (1) for details)
(C*)

Experiment/Material Applications:
The specific reasons why each of the individual sample materials was chosen were not presented in the available publications.

The thin oxide films were used as containers on some samples because of their ability to adhere to the molten material. This adherence was expected to prevent the onset of liquid surface movements which can cause convection in the bulk molten liquid. See Sprenger, TEXUS 1 for further discussion of using thin oxide films as containers.

References/Applicable Publications:
(1) Barbieri, F., Patuelli, C., Gonndi[sic], P., and Montanari, R.: Melting and Solidification in 0-g of Sintered Alloys-Experiments ES 311 A & B - 'Bubble Reinforced Materials'. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 101-107.

(2) Barbieri, F., Patuellim, C., Gondi, P., and Montanari, R.: Experiment ES-311 Bubble Reinforced Materials. Earth Oriented Applications of Space Technology, Vol. 5, Nos. 1-2, 1985, pp. 57-62.

(3) Castellani, L., Gondi, P., Barbieri, F., and Costa, N.: Dispersed Gaseous and Solid Phases in Molten and Solidified Cu. In Proceedings of the 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142 (June 1979), pp. 81-87. (pre-Spacelab flight; microgravity source unknown)

(4) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication (acceleration measurements on Spacelab 1)

(5) Whittmann, K.: The Isothermal Heating Facility. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 49-54. (IHF facility)

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Experiment Origin: France
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, ESA Materials Science Double Rack (MSDR)
Processing Facility: Low Temperature Gradient Heating Facility (GHF) Furnace
Builder of Processing Facility: CNES/Centre d'Etudes Nucléaires, Grenoble, France

Experiment:
Solidification of Al-Zn Vapor Emulsion (1ES316)

In situ composites are materials in which a minority component is formed by a physico-chemical reaction from a homogeneous liquid. Gravity-related phenomena (e.g., buoyancy, sedimentation, convection) can degrade the regularity of the resultant microstructure. Therefore, processing these materials under low-gravity conditions may result in a more homogeneous structure.

This Spacelab 1 experiment was the second in a series of investigations designed by Potard et al. to study low-gravity directional solidification (see Potard, SPAR 9 (Chapter 17)). The specific objectives of the study were to (1) study the directional solidification of an Al-Zn vapor emulsion and (2) investigate a new method of creating an in situ composite containing regularly dispersed bubbles. The directional solidification of an Al-Zn alloy under controlled zinc partial pressure and thermal profiles can result in a destabilization of the interfacial liquid zone. This destabilization gives rise to (1) vapor phase nucleation and growth and (2) a corresponding interaction of the formed bubbles with the moving solidification front.

During the mission, six Al-Zn samples (5, 2.5, or 1 at.% Zn) were processed in the Spacelab Gradient Heating Facility (GHF). "Each sample is contained in tubular crucible with a free meniscus maintained in the hot part of the samples. [The] Pressure drop is obtained by using a double wall crucible which allows to [sic]

decrease the Zinc vapour pressure at the free meniscus under its equilibrium value...." (1, p. 122) Since the creation of a vapor phase should be restricted to the solid/liquid interface, (1) a SiC crucible and (2) a high vacuum in the sealed capsule (which surrounded the crucible) were employed. (Ground tests had illustrated that SiC was wetted by Al.) Thermocouples were included in the cartridges for thermal data acquisition. Corresponding ground-based experiments were performed for comparison.

Post-flight x-ray analysis of the flight samples revealed a non-wetting of the Al to the SiC. Also, a 3 to 5 mm movement of the samples was reported. The non-wetting behavior, which was not observed during ground experiments, was attributed to the lack of gravitational forces which cause the liquid melt to contact the crucible walls. It also appeared that a vaporization of Zn at the melt/SiC interface occurred, which allowed the movement. As the thermal field developed, the pressure attempted to equilibrate. Therefore, a higher pressure existed at the cold end of the samples than above the free meniscus. It was reported that that an acceleration accident may have occurred which could have resulted in sample movement.

Metallographic examination of the 5 at.% flight-processed material (longitudinal cut, Keller's etch) revealed a large grain structure in the first solidified portion which became an elongated grain structure in the latter solidified portion. The last quarter of the sample contained numerous interconnected cavities. Overall, the sample exhibited no dendritic structure. However, the corresponding 1-g processed sample was clearly dendritic (same etching technique).

Examination of the 2.5 at.% flight-processed material (same metallographic technique) revealed three grain boundaries at the beginning of solidification "...which gives one single boundary oriented parallel to the sample axis after a certain distance...." (1, p. 124) Large dendrites (oriented parallel to sample axis) with voids between the secondary arms were located in the last portion of the sample. The 1-g processed sample showed new elongated grains oriented parallel to the sample axis direction. However, no dendrites were visible using etching techniques similar to those for the low-gravity material.

No discussion of the results from the Al-1 at.% Zn sample was provided in Reference (1).

Some of the conclusions of the study included:

(1) The microstructure of the 5 at.% alloy (no visible dendrites) was caused by the significant macrosegregation which occurred in the sample.

(2) The change in microstructure in the 5 at.% Zn sample may indicate the presence of convective currents within the melt.

It was surmised that the objective of the investigation, which was to create an aluminum alloy with a homogeneous bubble dispersion "...was only partially achieved." (1, p. 125)

"The main reason for this is that wetting was not obtained in space as well [as] it was on [the] ground. This will be studied for further interpretation.

"Nevertheless other features appearing in the samples are unexplained and probably linked to the particular nature of this kind of... [alloy] containing [a] high vapour pressure component." (1, p. 125)

See Reference (1) for further details.

Key Words: Composites with Gases, Emulsion, Metallic Matrix, Two-Phase System, Binary Systems, Metals and Alloys, Melt and Solidification, Directional Solidification, Thermal Gradient, Bubble Formation, Vaporization, Bubble Dispersion, Bubble Nucleation, Bubble Growth, Buoyancy Effects, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Reaction Kinetics, Buoyancy Effects, Sedimentation, Convection, Macrosegregation, Interface Stability, Free Surface, Free Surface Shape, Meniscus Shape, Solidification Front Physics, Sample Microstructure, Dendritic Structure, Dendrites, Dendritic Arm Spacing, Voids, Cavity, Grain Structure, Non-Wetting of Container, Wetting, Acceleration Effects, Vacuum

Number of Samples: six
Sample Materials: aluminum-zinc samples, compositions were listed as 5, 2.5 and 1 at.% of zinc
(Al*Zn*)
Container Materials: silicon carbide layer deposited on graphite
(Si*C*)

Experiment/Material Applications:
The specific reason why the Al-Zn alloys were selected for this experiment was not detailed in the available publications.

References/Applicable Publications:
(1) Potard, C. and Morgand, P.: Directional Solidification of a Vapour Emulsion Aluminum-Zinc in Microgravity. In Proceedings of the 5th Symposium on Materials Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 121-125. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48, Teledyne Brown Engineering Publication. (acceleration measurements on Spacelab 1)

(3) Input received from Experiment Investigator, July 1989 and August 1993.

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Experiment Origin: USA
Mission: STS Launch #24, STS-032, (STS 61-C, Columbia)
Launch Date/Expt Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment, Shuttle Student Involvement Program (SSIP), STS Middeck Experiment (middeck locker)
Processing Facility: Heating crucible with piston/needle assembly to initiate air bubbles and water quenching
Builder of Processing Facility: Unknown

Experiment:
Honeycombed Structures: Creation of Metallic Foam (SE83-6)

The objective of this shuttle experiment was to "...investigate the feasibility of creating a low density, high strength metal casting by injecting compressed air bubbles at specific intervals into a liquid alloy." (2, p. 1) It was expected that in the reduced gravity environment, the bubbles would not be affected by buoyancy forces, and an alloy structure superior to Earth-produced structures might be produced.

During the shuttle flight, a cerrelow 136 sample (a lead and bismuth alloy) was to be melted, injected with air, and then solidified. <Note: Reference (1) indicated that the sample was to be injected with argon.>

Reportedly, "The experiment was unsuccessful.... The metal failed to melt completely. Therefore no bubble could be introduced into the melt." (2, p. 2)

Key Words: Composites with Solid Particles, Metals and Alloys, Ternary Systems, Two-Phase System, Melt and Solidification, Casting, Gas Injection, Bubble Injection, Bubble Dispersion, Liquid/Gas Dispersion, Liquid/Gas Interface, Porosity, Solid/Liquid Interface, Buoyancy Effects, Metallic Foams, Surface Tension, Piston System, Material Strength, Incomplete Sample Processing

Number of Samples: one
Sample Materials: cerrelow (lead and bismuth) injected with air (Pb*Bi*)
Container Materials: unknown

Experiment/Material Applications:
"The research could provide helpful data on the future production, in space, of foamed metals useful for many applications both in space and on the ground." (2, p. 1)

References/Applicable Publications:
(1) Space Shuttle Mission 61-C, NASA Press Kit, December 1985, p. 20. (preflight)

(2) Bubble Injection as a Alternative to Honeycombing. In Shuttle Student Involvement Program (SSIP) Final Reports of Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989, 3 pp. (post-flight)

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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Two-chambered mixing apparatus employing a motor-driven stirring propeller
Builder of Processing Facility: Dr. F. C. Wessling, Consortium for Materials Development in Space at the University of Alabama in Huntsville

Experiment:
Foam Formation

The non-uniform cell-size of foams produced on Earth are due in part to (1) hydrostatic pressure acting on the foam and (2) bubble sedimentation (and associated streaming). A more uniform cell size is expected to result when foams are produced in the low-gravity environment. In such an environment, the gravity-driven phenomenon that adversely affects the cell size should be reduced.

The objectives of this Consort 1 experiment were to (1) form a polymer-gas foam (polyurethane) and (2) determine if the properties of the low-gravity produced foam were significantly different from terrestrially-produced foams.

Chemicals used to produce the foaming reaction were stored in two chambers within the experimental apparatus. The first chamber contained a polyol solution; the second chamber contained diisocyanate. During the rocket flight (just prior to the onset of the low gravity period), a piston located in the first chamber was moved, expanding the available volume in the chamber. The diisocyanate was injected into the expanded chamber. The chemicals (diisocyanate plus polyol) were then mixed for 19 seconds by an electrically driven, motorized propeller. At this point of the experiment, the low-gravity phase of the rocket had been reached. During the following 11 seconds, stirring continued and the mixture was ejected through a flow restriction orifice and

into an exit funnel. (The purpose of the orifice was to slow the speed of the ejected fluid.) A screen at the end of the funnel outlet provided a surface for foam formation.

Photographs of the mixture as it exited the funnel were taken by a camera placed directly opposite the orifice. By strategically placing mirrors about the experiment payload, images of the exiting mixture from other angles were documented on the film.

It was expected that the following time-line would result: "Chlorofluorocarbon would begin to come out of the solution at the cream time, which would occur 28-37 s after mixing. Within 10 s of the cream time, the material would begin to expand. A 10-20 fold volume increase would be achieved 180-200 s after initial mixing. The foam would reach its final state after 120-180... [seconds] of additional cure." (1, p. 324)

Post-flight analysis of the payload illustrated that the experimental apparatus operated as designed. A spherical ball of foam (approximately 17 cm in diameter) formed and remained attached to the funnel screen. It was presumed that surface tension effects had dominated (as expected) to yield the spheroidal shape of the foam. The expected timeline as detailed above progressed essentially as outlined. It was reported that "...the cream time and the expansion time do not appear to be significantly affected by low gravity." (1, p. 327)

A thermistor placed in front of the funnel indicated that the temperature varied from 20 ^{o}C to 48 ^{o}C during the 800-s rocket flight. Many temperature fluctuations throughout the experiment were of interest and are discussed in Reference (1), p. 328. Perhaps one of the most interesting measurements "...indicated that the blowing agent of the foam was above its saturation temperature when the foam mixture exited the funnel... the blowing agent had approximately 6 ^{o}C of superheat when it exited the funnel." (1, p. 328)

Comparisons were made of cross sections of polyurethane foam formed on Consort 1 and on Earth. In both types of samples, pore size and shape varied throughout the section. Because the pore size was variable, testing of the mechanical and thermal properties were not completed. Such tests require uniform pore distributions. It was noted that "...the cells of the foam made... [on Consort 1] tended to be spherical in shape. Those formed on Earth tended to be elongated. The entire foam mass was spherical when formed... [on Consort 1] and shaped like a large pancake when formed on Earth without a container and in the shape of its container otherwise." (1, p. 328) "The exact cause for the various size pores... has not yet been determined but may be due to the blowing agent being superheated when stirred." (1, p. 329)

It was further noted that the quality of the Consort-produced foam "...does not appear to be the same as industrial quality foam. However this is not inherently due to processing in low gravity but due to possible shortcomings in the processing equipment." (1, p. 329) <Note: The information in Reference (1) did not detail if the quality of the Consort-produced foam was superior or inferior to the industrial-quality foam.>

Key Words: Composites with Gases, Foams, Foam Stability, Polymer-Gas Foams, Multiphase Media, Bubble Formation, Bubble Dispersion, Bubble Sedimentation, Gas Formation, Porosity, Pore Size, Sphericity, Hydrostatic Pressure, Surface Tension, Surfactants, Liquid Injection, Liquid Expulsion Through a Small Orifice, Liquid Mixing, Stirring of Components, Buoyancy Effects, Liquid/Gas Dispersion, Liquid/Gas Interface, Solid/Liquid Interface, Catalysts, Reactant Solutions, Reaction Kinetics, Saturation, Superheating, Material Strength, Piston System, Space Structures

Number of Samples: one
Sample Material: The polyurethane foam which was created was formulated from a "...two part mixture including a solution of sucrose-based polyol, catalyst, surfactant, and fluorocarbon blowing agent in one chamber and an oligometric diisocyanate in another chamber.... " (1, p. 324) These proportions were used: (1) polyol - 100 g, (2) catalyst - 2.0 g, (3) surfactant - 1.5 g, (4) blowing agent - 3.4 to 3.6 g, and (5) diisocyanate - 92.5 g. (See Reference (1), p. 324 for more details.)
Container Materials: not applicable

Experiment/Material Applications:
"Cellular materials, formed by incorporating gas bubbles in a polymetric matrix, often have useful thermal and mechanical properties. Rigid polyurethane foams are commonly recognized as outstanding materials for insulation applications....

"A number of important structural features are required for a foam to have useful physical properties. A good foam will have cells with diameters of 200 to 1700 μm. Foams with a closed cell configuration are used for insulation and have higher mechanical strength. Foams with densities less than 0.032/g/ml cannot support a predominately closed cell structure as the thin cell walls will rupture easily. It has been assumed that gravity influences

the cell shape of a rigid polyurethane foam. Our ultimate goal is to examine the process under low-g conditions where nearly-perfect small cells may be formed." (2, p. 45)

"Annual usage of polyurethane foams is over 600 million lb worldwide. In addition to potential uses as an insulating material, space applications proposed include uses for micrometeroid barriers... and structural material for lightweight structures in space. Also, understanding the mechanisms of foam formation may have large economic impacts in thermal insulation values. Being able to create foams in the space environment may lead to building structures in space, such as habitats on the moon, re-entry vehicles, or large structures." (1, p. 324)

References/Applicable Publications:

(1) Wessling, F. C., McManus, S. P., Matthews, J., and Patel, D.: Foam Formation in Low Gravity. Journal of Spacecraft and Rockets, Volume 27, No. 3, May-June 1990, pp. 324-329. (post-flight)

(2) Foam Formation. In Consortium for Materials Development in Space, The University of Alabama in Huntsville, Annual Report, Technical Section, October 1, 1988-September 30, 1989, pp. 45-46. (post-flight)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. 40th Congress of the International Astronautical Federation, October 7-12, 1989, Màlaga, Spain, IAF-89-439, 10 pp. (post-flight)

(4) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight)

(5) Input received from Experiment Investigators, June 1991 and July 1993.

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CHAPTER 5

COMPOSITES WITH SOLID PARTICLES

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Experiment Origin: USA
Mission: Apollo 14
Launch Date/Expt. Date: February 1971
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Apollo Command Module Payload, Science Demonstration
Processing Facility: An electric heater which accepted sealed, sample-material capsules
Builder of Processing Facility: Heater and solidification capsules were provided to investigators by NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Composite Casting-Part I: Powder Metallurgy Samples

<Note: A total of eleven samples were processed during the Composite Casting Experiment. The evaluation of three of those samples (denoted by the investigators as "Part I" of the experiment) is discussed here. Discussion of the other eight samples can be found under Peters, Apollo 14 (Chapter 5); Reger, Apollo 14 (Chapter 17); Steurer, Apollo 14 (Chapter 5).>

The objective of this Apollo 14 composite casting experiment (Part I) was to examine the potential for forming unique metal matrix composites in the reduced gravity environment.

Prior to the mission, three sample capsules were prepared for the experiment. Each of the capsules contained InBi and a distribution of either tungsten particles or boron carbide particles. During the mission, each capsule was placed into an electric heater and warmed until melting of the sample took place. Solidification occurred when the heater and sample were placed in a heat sink.

Cornell Aeronautical Laboratory conducted post-flight evaluation of the three samples and compared them to Earth-processed control samples.

The first sample had been processed to evaluate the distribution of higher density, spherical copper-coated tungsten particles in a lower density InBi eutectic powder matrix. Redistribution of the particles occurred in both ground and flight samples. Reportedly, the flight samples exhibited a more uniform tungsten distribution.

The second sample had been processed to evaluate the distribution of lower density spherical copper-coated boron carbide particles and gas bubbles in the higher density, indium bismuth eutectic powder matrix. Significant segregation was expected in the ground sample, while a more homogeneous distribution was expected in the flight sample. Reportedly, results were inconclusive because the space-processed powder compacts did not melt.

The third sample had been processed to evaluate the distribution of copper-coated tungsten particles into molten InBi eutectic. Reportedly, prior to the mission, "Approximately 105 g of copper-coated spherical tungsten particles (100 μ diam.) were placed in a preheated capsule, a tungsten mixing pellet added and then 100 g of In-Bi eutectic poured into the capsule." (3, p. 17). During both the flight and ground experiments, the heater was shaken to disperse the particles within the matrix material. Because the flight sample was also shaken by "RCS" firings during sample cooling, "...it was melted and shaken in space more than once.... The total time this sample was molten and shaken is not known...." (1, p. 56) It was noted that "When the [ground] control sample was opened, large quantities of the microspheres fell out of the container, and cutting the sample in half released more of the microspheres. The copper coated microspheres had oxidized prior to filling the capsule and were therefore not wetted by the molten indium bismuth. They aggregated and did not disperse through the specimen." (1, p. 56) Similar oxidation of the microspheres seems to have occurred in the flight sample, although a more homogeneous distribution of tungsten particles was observed in the space processed sample.

A detailed analysis of each of the three samples is presented in Reference (2).

Key Words: Composites with Solid Particles, Composites With Gases, Ternary Systems, Powder Metallurgy, Metals, Eutectics, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Homogeneous Dispersion, Particle Distribution, Particle Aggregation, Coated Particles, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Liquid/Gas Dispersion, Solid/Gas Dispersion, Solid/Liquid Interface, Liquid/Gas Interface, Bubbles, Segregation, Density Difference, Separation of Components, Stirring of Components, Thermal Convection, Wetting, Particle Wetting, Oxidation, Liquid Phase Sintering, Incomplete Sample Processing

Number of Samples: three
Sample Materials: Sample 1: 70% indium bismuth eutectic powder with 30% copper-coated tungsten spheres; Sample 2: 70% indium bismuth eutectic powder with copper-coated boron carbide (B_4C) spheres; Sample 3: 70% indium bismuth eutectic metal matrix with 30% copper-coated tungsten spheres (100 μ diam).
(In*Bi*, W*, Cu*, B*C*)
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
The first two samples were processed to demonstrate the reduced gravity distribution of particles in a powder metal matrix. If a more homogeneous distribution was observed in the flight sample, liquid phase sintering in low gravity may be possible. The third sample was processed to evaluate the possibilities of creating unique metal matrix composites in the reduced gravity environment.

References/Applicable Publications:
(1) Yates, I. C.: Apollo 14 Composite Casting Demonstration. In Process Engineering Research at MSFC, Research Achievements Review, Vol. IV, Report No. 7, Marshall Space Flight Center, Alabama, NASA TM X-64723, February 1973. (post-flight)

(2) Fabiniak, R. C. and Fabiniak, T. J.: Test and Evaluation of Apollo 14 Composite Casting Demonstration Specimens and Flight and Control Samples. KE-3101-D-1, Cornell Aeronautical Laboratory, Inc. of Cornell University, Buffalo, New York, September 1971. (post-flight)

(3) Yates, I. C., Jr.: Apollo 14 Composite Casting Demonstration-Final Report. NASA TM X-64641, October 1971.

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Experiment Origin: USA
Mission: Apollo 14
Launch Date/Expt. Date: February 1971
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Apollo Command Module Payload
Processing Facility: An electric heater which accepted sealed, sample-material capsules
Builder of Processing Facility: Heater and solidification capsules were provided to investigators by NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Composite Casting-Part II: Eutectic Solidification Samples

<Note: A total of eleven samples were processed during the Composite Casting Experiment. The evaluation of three of those samples (denoted by the investigators as "Part II" of the experiment) is discussed here. Discussion of the other eight samples can be found under Fabiniak, Apollo 14 (Chapter 5); Reger, Apollo 14 (Chapter 17); Steurer, Apollo 14 (Chapter 5).>

The objective of this Apollo 14 composite casting experiment (Part II) was to examine the potential for forming unique metal-matrix composites in the reduced gravity environment.

The experiment employed three sample capsules. Each of the capsules held an InBi eutectic prepared with a distribution of solids and/or gas. During the mission, each sample was placed in an electric heater and warmed until melting of the contents took place. The heater was then shaken by hand to disperse solids and/or gas within the matrix material. Solidification occurred when the heater and sample were placed into a heat sink.

Arthur D. Little, Inc. conducted post-flight evaluation of three of the returned samples and compared them to Earth processed control samples.

The first sample had been processed to evaluate the distribution of copper-coated silicon carbide whiskers and argon gas bubbles within the metal matrix InBi. Examination of the flight sample indicated that it exhibited a more uniform microstructure and distribution of gas pores than the similarly processed terrestrial sample.

The second sample had been processed to evaluate the distribution of argon gas in the metal matrix InBi. During ground-based sample preparation, a steel mixing screen had been inserted in the molten InBi to promote gas foaming when the sample was later shaken during processing. Examination of the flight sample indicated that some gas porosity was present, although in the strict sense there was no foam. The ground-based control sample indicated no foam or gas porosity.

The third sample had been processed to evaluate the distribution of argon gas and copper-coated spherical tungsten particles in the InBi eutectic. Examination of the flight sample indicated that (1) the argon had leaked from the capsule (presumably during a welding operation), (2) tungsten spheres were distributed in the bulk of the sample, and (3) the microstructure had large areas of aligned fine lamellar eutectic. In contrast, examination of the ground-based control sample indicated that (1) no tungsten spheres were distributed in the bulk of the sample, and (2) solidification did not occur in a plane interface normal to the axis of the sample.

A detailed discussion of the analysis of each sample can be found in Reference (2).

Key Words: Composites with Solid Particles, Composites with Gases, Ternary Systems, Eutectics, Lamellar Eutectics, Metals, Metallic Matrix, Metallic Foams, Melt and Solidification, Casting, Segregation, Whiskers, Density Difference, Particle Dispersion, Coated Particles, Bubble Dispersion, Homogeneous Dispersion, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Liquid/Gas Dispersion, Solid/Liquid Interface, Liquid/Gas Interface, Stirring of Components, Bubbles, Foams, Porosity, Interface Physics, Planar Solidification Interface, Sample Microstructure, Gas Leakage

Number of Samples: three

Sample Materials: (1) InBi Eutectic with uniform distribution of a solid (SiC whiskers) and a gas (argon) (75% InBi with SiC, 25% Ar); (2) InBi eutectic with a distribution of argon gas (Stainless steel screen embedded to promote foaming) (75% InBi, 25% Ar); (3) InBi with dispersion of argon and copper-coated tungsten spheres (75% InBi with W, 25% void)
(In*Bi*, Si*C*, Ar*, W*)
Container Materials: aluminum
(Al*)

Experiment/Material Applications:
Because this experiment was among the first to explore the area of metal solidification in space, its goals were to (1) examine space solidification phenomena, and (2) discern if mixtures of solids, liquids, and gases of different densities would disperse uniformly thus resulting in unique composite materials.

References/Applicable Publications:
(1) Yates, I. C.: Apollo 14 Composite Casting Demonstration. In Process Engineering Research at MSFC, Research Achievements Review, Vol. IV, Report No. 7, Marshall Space Flight Center, Alabama, NASA TM X-64723, February 1973. (post-flight)

(2) Peters, E., et al.: Apollo 14 Composite Casting Demonstration. NASA CR-61369 (Arthur D. Little, Inc.), NASA Marshall Space Flight Center, Alabama, August 1971. (post-flight)

(3) Yates, I. C., Jr.: Apollo 14 Composite Casting Demonstration-Final Report. NASA TM X-64641, October 1971. (post-flight)

(4) NASA Tech Brief, Marshall Space Flight Center, B72-10266, June 1972. (post-flight)

(5) Input received from Principal Investigator E. T. Peters, April 1990 and August 1993.

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Experiment Origin: USA
Mission: Apollo 14
Launch Date/Expt. Date: February 1971
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Apollo Command Module Payload
Processing Facility: An electric heater which accepted sealed, sample-material capsules
Builder of Processing Facility: Heater and solidification capsules were provided to investigators by NASA, Marshall Space Center, Huntsville, Alabama

Experiment:
Composite Casting-Part IV: Fiber Reinforced Matrix Samples

<Note: A total of eleven samples were processed during the Composite Casting Experiment. The evaluation of two of those samples (denoted by the investigators as "Part IV" of the experiment) is discussed here. Discussion of the other nine samples can be found under Fabiniak, Apollo 14 (Chapter 5); Peters, Apollo 14 (Chapter 5); Reger, Apollo 14 (Chapter 17).>

The specific objective of this Apollo 14 composite casting experiment (Part IV) was to examine the potential for forming unique fiber reinforced materials in the reduced gravity environment. Other tasks mentioned in Reference (5) included (1) evaluation of potential processes for space manufacturing, (2) definition of effective processing concepts and products, (3) identification of potential process development programs, (4) evaluation and selection of suitable Apollo 14 sample materials, (5) definition of Apollo 14 in-flight processing procedures, (6) preparation of preprocessed Apollo 14 materials (and delivery to NASA), and (7) evaluation of the space processed sample after return from the Apollo 14 flight.

Prior to the mission, two sample capsules were prepared for the experiment. Each of the capsules contained a matrix material and coated beryllium copper fibers. During the mission, each capsule was placed in an electric heater and warmed until melting of the

sample took place. The heater was then shaken by hand to mix the materials. Solidification took place when the heater and sample were placed in a heat sink.

General Dynamics conducted the post-flight evaluation of the two samples and compared them to similar Earth-processed control samples.

The first sample was processed to demonstrate the dispersion of beryllium copper fibers and argon gas bubbles in an indium-bismuth eutectic metal matrix. Post-flight evaluation of the flight sample indicated that most of the gas had unexpectedly leaked from the material during ground-based preparation of the capsule. However, examinations of flight sample indicated that the fibers in the material were dispersed over the entire cross-section. Any nonuniformities of the dispersion were attributed to the primitive mixing method employed. In contrast, examinations of the ground-based sample indicated gravity-induced segregation "...even though the density between fibers and matrix was only 1%, the bulk of the fibers settled in the lower section, while the upper section was virtually free of fibers." (2, p. 7-8) Reportedly, there was a tendency for fiber coagulation at gas bubble interfaces in the flight sample. Further, there was an inconclusive tendency for fibers to align themselves in the reduced gravity environment which indicated possible controlled fiber orientation.

The second sample was processed to demonstrate the dispersion of cerrobend-coated beryllium copper fibers and argon gas bubbles in paraffin. Post-flight evaluation of the sample indicated that most of the gas had unexpectedly leaked from the material during ground-based preparation of the capsule. Further, "At the time of sample preparation, only coated fibers were available for the flight sample.... Since the processing temperature was higher than the melting temperature of the coating, the coating melted during processing, causing the fibers to literally solder themselves into one single block of paraffin-fiber composite, while the remaining part of the sample was pure paraffin.... Consequently, the objectives of fiber dispersion and fiber-bubble interaction were not achieved." (2, p. 7-20) It was reported, however, that fairly stable dispersion of gas bubbles was achieved in the paraffin flight sample with only a small amount of gas (low bubble density). In contrast, the similar ground-based sample exhibited no bubble dispersion (all the bubbles had been removed by buoyancy forces).

Key Words: Composites with Solid Particles, Composites with Gases, Ternary Systems, Reinforced Materials, Eutectics, Metals, Metallic Matrix, Melt and Solidification, Casting, Buoyancy Forces, Segregation, Density Difference, Buoyancy Forces, Coagulation, Particle Dispersion, Coated Particles, Fiber Dispersions, Coated Fibers, Bubble Dispersion, Homogeneous Dispersion, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Liquid/Gas Dispersion, Solid/Liquid Interface, Liquid/Gas Interface, Stirring of Components, Bubbles, Interface Physics, Sample Microstructure, Material Strength, Gas Leakage, Processing Difficulties

Number of Samples: two
Sample Materials: (1) Indium bismuth eutectic alloy with coated beryllium copper fibers (75% InBi with BeCu, 25% void due to gas leak); (2) paraffin with cerrobend-coated beryllium copper fibers (75% Paraffin with BeCu, 25% void due to gas leak).
(In*Bi*Be*Cu*)
Container Materials: Aluminum. A "non-stick coating" was thought to have been applied to the inside surfaces.
(Al*)

Experiment/Material Applications:
Space processing initiatives such as those investigated here may result in (1) high performance composite materials and cast components with uniform and stable dispersions of reinforcement fibers, (2) fiber reinforced foam materials or cast components with high stiffness and strength-to-weight ratios, (3) high performance composite materials and components with controlled fiber orientation, and (4) metal foam materials.

References/Applicable Publications:
(1) Yates, I. C.: Apollo 14 Composite Casting Demonstration. In Process Engineering Research at MSFC, Research Achievements Review, Vol. IV, Report No. 7, Marshall Space Flight Center, Alabama, NASA TM X-64723, February 1973. (post-flight)

(2) Steurer, W. H. and Kaye, S.: Preparation and Evaluation of Apollo 14 Composite Experiments. NASA CR-61368 (General Dynamics Convair), NASA Marshall Space Flight Center, Alabama, August 1971. (post-flight) (This report can be found on microfiche at the General Dynamics/Convair Division Engineering Library.)

(3) Kaye, S. and Raat, J.: Low Gravity Dispersion of Solids in Liquid Metals. In Proceedings of the Third Space Processing Symposium on Skylab Results, April 30-May 1, 1974, Marshall Space Flight Center, Alabama, Vol. II, June 1974, pp. 857-885. (related discussion)

(4) Yates, I. C., Jr.: Apollo 14 Composite Casting Demonstration-Final Report. NASA TM X-64641, October 1971.

(5) Input received from J. A. Pardubsky, August 1989.

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Experiment Origin: Belgium
Mission: TEXUS 6
Launch Date/Expt. Date: May 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2: Multipurpose Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Particle Composites-- Dispersion Alloys

Metallic composite materials, such as those with (1) a metal matrix and (2) ceramic or metal particles, may possess important properties (e.g., considerable thermal conductivity, reasonable high temperature strength, and high abrasive wear resistance). Typically, these materials are manufactured using expensive and complicated powder metallurgical techniques. An easier manufacturing method would be the direct addition of the particles to a melt, followed by casting. However, because of gravity-induced effects (e.g., sedimentation, thermal convection), the desired distribution of the particles would not always be obtained. A better understanding of the fluid phenomenon which occurs during metallic composite solidification may lead to (1) improved casting methods and (2) superior composite materials.

This TEXUS 6 experiment was the second in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab SL-3, Chapter 4: "Composites with Gases."). The specific objectives of the experiment were to (1) identify the forces which cause displacement of particles within a melt and (2) obtain a homogeneous distribution of SiC particles well-bound to a Cu matrix after processing. Of particular interest were the interfacial properties of the phases present: (1) the liquid phase of the metal matrix, (2) the gaseous phases, and (3) the solid phases of the (a) dispersed particles, (b) solidifying matrix, and (c) crucible wall material.

Copper samples containing angularly-shaped, 125-160 micron diameter SiC particles were selected for the experiment. Prior to launch, three such samples of Cu-0.25 vol.% SiC were prepared by a mechanical mixing and hot extrusion process. Two of the samples were enclosed in TMZ-molybdenum alloy crucibles (well-

wetted by liquid copper) and one was enclosed in a graphite crucible (poorly wetted by liquid copper). All three crucibles were then enclosed in a single TMZ cartridge. Three thermoelements were fixed on the outside surface of the cartridge (one at each end and one in the middle). The cartridge was placed in a multipurpose furnace housed within the TEXUS Experiment Module TEM 01-2.

Just prior to launch, the samples were preheated to 850 ^{o}C. Once the low-gravity period of the flight was achieved, rapid heating and melting of the sample occurred followed by solidification. Similar reference samples were processed on Earth using the same time-temperature profile.

Post-flight examination of the flight thermal data revealed that melting and solidification occurred under low-gravity conditions. (The samples had been molten for about 3 minutes.) However, the samples were subjected to an unintended thermal gradient of 50 ^{o}C/cm. Examination of the space-processed samples revealed that "...liquid copper from two samples... [was] sucked out of the TMZ-crucible by capillary action, resulting in rather large gas holes and inducing additional flow currents in the liquid...." (Reference (3)) Reportedly, such capillary action did not occur in the graphite crucible and particles were found at the periphery of the Cu matrix.

Further examination of all the samples (1-g and low-g) indicated that the particles separated from the matrix during the melting phase of the experiment. During heating, "...extensive dissolution of SiC and diffusion of Si in the copper took place. This alloying effect lowered the melting point of the copper matrix around the SiC particles. The volume expansion due to the partial melting of the alloyed zones caused an internal pressure; also, convection currents were induced by concentration differences in the liquid. In a further phase, these liquid zones became interconnected leading to exudation of all SiC particles, even before the matrix was completely melted." (2, p. 66) This mechanism resulted in SiC particles collecting in gas holes (in the wetted TMZ crucible) or at crucible/matrix interface (in the non-wetted graphite crucible).

Key Words: Composites with Solid Particles, Model Materials, Dispersion Alloys, Powder Metallurgy, Binary Systems, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Homogeneous Dispersion, Solid/Liquid Interface, Sedimentation, Interface Physics, Inclusion and/or Rejection of Particles, Dissolution, Thermal Gradient, Thermal Convection, Diffusion, Diffusive Mass Transfer, Crucible Effects, Wetting of Container, Non-Wetting of Container, Liquid Leakage, Volume Expansion, Capillary Forces, Wetting, Free Surface, Solutal Gradients, Marangoni Convection, Surface Energy, Separation of Components, Material Strength, Furnace Malfunction, Processing Difficulties

Number of Samples: three
Sample Materials: copper with silicon carbide particles, Cu-0.25 vol.% SiC particles
(Cu*Si*C*)
Container Material: two samples in TMZ-molybdenum alloy crucibles and one sample in a graphite crucible
(Mo*, C*)

Experiment/Material Applications:
The Cu-SiC material was selected for this investigation because the limited capabilities of the experimental hardware allowed only the use of model systems.

References/Applicable Publications:
(1) Froyen, L. and Deruyttere, A.: Metallic Composite Materials and Microgravity. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, p. 31. (post-flight results)

(2) Deruyttere, A. and Froyen, L.: Melting and Solidification of Metallic Composites. In Proc. RIT/ESA/SSC Workshop "The Effect of Gravity on the Solidification of Immiscible Alloys," Stockholm, January 18-20, 1984, ESA SP-219, pp. 65-68. (post-flight)

(3) Input received from Principal Investigator A. Deruyttere, June 1988.

(4) Deruyttere, A. and Froyen, L.: Metallic Composite Materials with Particles. To Be Published by ESA. (post-flight; received from Principal Investigator A. Deruyttere, June 1988.) <Note: The current publication status of this document is unclear.>

(5) Deruyttere, A., Froyen, L., and De Bondt, S.: Melting and Solidification of Metallic Composites in Space. In Advances in Space Research, Vol. 6, Number 5, 1986, pp. 101-110. (post-flight; TEXUS 6, TEXUS 7, TEXUS 9, Spacelab 1, and Spacelab D1 results)

(6) Input received from Principal Investigator L. Froyen, August 1993.

(7) Metallic Composites with Particles. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 296-297. (post-flight)

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Principal Investigator(s): Deruyttere, A. (1), Froyen, L. (2)
Co-Investigator(s): None
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Experiment Origin: Belgium
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2: Multipurpose Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Particle Composites - Dispersion Alloys

This TEXUS 7 experiment was the third in a series of investigation designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab SL-3 (Chapter 4), TEXUS 6). The specific objective of the experiment was to identify the forces which cause displacement of particles within a melt. Of particular interest were the interfacial properties of the phases present: (1) the liquid phase of the metal matrix, (2) the gaseous phases, and (3) the solid phases of the (a) dispersed particles, (b) solidifying matrix, and (c) crucible wall material.

Prior to the rocket launch, three Cu-dispersed particle samples were prepared. Sample 1 was a copper sample with 32-45 micron diameter SiO_2 particles. The Cu-0.3 wt.% SiO_2 sample was contained in a TMZ alloy crucible (well-wetted by the copper melt). The TMZ crucible was configured with a non-wetted graphite ring at the top, which reduced the free melt surface. Sample 2 was a copper sample with 20-45 micron diameter W particles. The Cu-30 wt.% W sample was contained in a pure copper shell. The copper shell prevented the interaction between the crucible wall and particles during sample melting. Sample 3 was the same as Sample 1 except it was contained in a non-wetted graphite crucible. (It was noted that tungsten particles are well-wetted by the copper matrix (wetting angle, theta, is approximately 60^{o}) while the copper does not wet the SiO_2 particles well (theta is approximately $140\text{-}170^{o}$).)

During the TEXUS 7 Mission, the TEXUS Experiment Module TEM 01-2 Multipurpose Furnace was used to process the samples. While under low-gravity conditions, the samples were (1) melted, (2) maintained in a molten state (for approximately 3 minutes), and (3) resolidified. Thermal data indicated that the samples were

slightly overheated (30 ^{o}C) during the mission.

Similar reference samples were also processed under 1-g conditions. It was reported that the experimental procedure used during these 1-g investigations was analogous to that of the TEXUS 6 experiment.

Post-flight examination of Sample 1 (low-gravity) revealed an almost complete retention of the SiO_2 particles with uniform distribution throughout most of the sample. In contrast, the 1-g-processed sample illustrated a dominant effect of Stokes sedimentation.

The SiO_2 particles in Sample 3 (low-gravity) were only partially retained in the matrix. The particles were either (1) agglomerated or (2) clinging to small gas holes. In the similarly processed 1-g sample, the particles had completely exuded to the top.

The W particles of Sample 2 (1-g) had completely lost their initial skeletal form, either floating to the top or settling on the bottom. Examination of the low-gravity processed sample indicated that the particles remained in their skeletal form, which moved as a whole over a small distance. This movement was attributed to residual accelerations during the TEXUS 7 flight.

It was reported that the main conclusion from the TEXUS 6 and TEXUS 7 experiments was that in a low-gravity environment, it is possible to maintain a fairly uniform dispersion of non-wetting particles in a matrix melted in a wetted crucible. Particle dispersion was not maintained in similar experiments on Earth and under low-gravity conditions when the materials were melted in a non-wetting crucible.

Key Words: Composites with Solid Particles, Model Materials, Dispersion Alloys, Binary Systems, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Particle Agglomeration, Solid/Liquid Dispersion, Solid/Liquid Interface, Solid/Liquid/Gas Dispersion, Homogeneous Dispersion, Sedimentation, Stokes Sedimentation, Interface Physics, Inclusion and/or Rejection of Particles, Crucible Effects, Wetting of Container, Non-Wetting of Container, Wetting, Particle Wetting, Surface Energy, Free Surface, Material Interaction with Containment Facility, Thermal Environment More Extreme than Predicted, Acceleration Effects, Buoyancy Effects

Number of Samples: three
Sample Materials: Sample 1: Cu with 0.3 wt.% SiO_2 particles; Sample 2: Cu with 30 wt.% W particles; Sample 3: Cu with 0.3 wt.% SiO_2 particles
(Cu*W*, Cu*Si*O*)
Container Materials: Sample 1: TMZ-molybdenum alloy; Sample 2: pure copper shell; Sample 3: graphite
(Mo*, Cu*)

Experiment/Material Applications:
See Deruyttere, TEXUS 6.

References/Applicable Publications:
(1) Froyen, L. and Deruyttere, A.: Metallic Composite Materials and Microgravity. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, p. 31. (preflight)

(2) Deruyttere, A. and Froyen, L.: Melting and Solidification of Metallic Composites. In Proc. RIT/ESA/SSC Workshop "The Effect of Gravity on the Solidification of Immiscible Alloys," Stockholm, January 18-20, 1984, ESA SP-219, pp. 65-68. (post-flight)

(3) Input received from Principal Investigator A. Deruyttere, June 1988.

(4) Deruyttere, A. and Froyen, L.: Metallic Composite Materials with Particles. To Be Published by ESA. (post-flight; received from Principal Investigator A. Deruyttere, June 1988.) <Note: The current publication status of this document is unclear.>

(5) Deruyttere, A., Froyen, L., and De Bondt, S.: Melting and Solidification of Metallic Composites in Space. In Advances in Space Research, Vol. 6, Number 5, 1986, pp. 101-110. (post-flight; TEXUS 6, TEXUS 7, TEXUS 9, Spacelab 1, and Spacelab D1 results)

(6) Froyen, L. and Deruyttere, A.: Melting and Solidification of Metal Matrix Composite Under Microgravity. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss, Elmau, November 5-7, 1984, ESA SP-222, pp. 69-78. (post-flight; TEXUS 7 and Spacelab 1 Results)

(7) Input received from Principal Investigator L. Froyen, August 1993.

(8) Metallic Composites with Particles. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 298-299. (post-flight)

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Experiment Origin: Belgium
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Melting and Solidification of Metallic Composites (1ES315)

This Spacelab 1 experiment was the fourth in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab SL-3 (Chapter 4), TEXUS 6, TEXUS 7). The specific objectives of this experiment were to (1) melt and solidify metal matrix composites, (2) investigate the mechanisms which cause a destabilization of the metallic suspensions, and (3) investigate the effect of interfacial phenomena on the preparation and properties of metallic composites.

Prior to the shuttle flight, six aluminum matrix samples and six copper matrix samples were prepared. Each of the copper matrix samples contained either alumina, tungsten, molybdenum, or silicon carbide particles. The aluminum matrix samples had the following specific additions: (a) Sample 1 contained approximately 7 wt.% rounded Al_2O_3 particles (0.1 to 0.5 microns), (b) Sample 2 contained 0.5 wt.% angular SiC particles (0 to 3.0 microns), (c) Sample 3 contained 0.2 wt.% angular SiC particles (0 to 15 microns), (d) Sample 4 contained 0.5 wt.% angular SiC particles (0 to 15 microns), (e) Sample 5 contained 0.2 wt.% angular SiC particles (125 to 160 microns), and (f) Sample 6 contained 0.5 wt.% angular SiC particles (125 to 160 microns).

Each aluminum matrix sample was prepared by (1) mixing (Al with SiC particles) or mechanical alloying (Al-Al_2O_3 sample), (2) cold pressing, (3) degassing, (4) extrusion (after heating in an inert atmosphere), and (5) machining to proper dimensions. The samples were enclosed in a single graphite crucible, which was contained (after degassing) in a tantalum cartridge. Three Pt/Pt-Rh thermocouples were also contained in the graphite crucible. (Reference (1) contains additional details of sample preparation, and further discusses the copper matrix samples.)

The six aluminum matrix samples and six copper matrix samples were to be melted and resolidified in the Spacelab 1 Isothermal Heating Facility (IHF). However, because of a "...malfunctioning of the... I.H.F... the second run with the copper matrix samples was cancelled." (1, p. 70) (The I.H.F. malfunction was attributed to a general electrical failure which could not be repaired by the shuttle crew.) Instead, only six aluminum matrix samples were processed.

During the flight experiment, the cartridge containing the aluminum matrix samples was heated to 800 ^{o}C and held at this temperature for 40 minutes. Helium gas was then introduced to solidify the samples. A cooling rate of 1 K/s resulted. Further cooling of the samples occurred at ambient temperature in a cooling chamber. <Note: The location of this cooling chamber is not known.>

Similar reference experiments were performed on Earth using nearly identical thermal conditions. Post-flight analysis of the samples included (1) radiography, (2) metallography, (3) Vickers macro-hardness tests (10 kg load, approximately 1 mm indentation), (4) tensile tests, (5) fracture surface examination, and (6) abrasive wear tests. Reportedly, hardness tests were only performed on Samples 1, 2, and 4 because gas holes (Sample 3) and large carbide particles (Samples 5 and 6) invalidated hardness measurements. Casting defects also caused premature fracture during tensile tests, resulting in inadequate evaluation of strength and ductility characteristics in both the 1-g and low-gravity processed samples.

Reportedly, the results and achievement of the experimental objectives were limited because the IHF malfunctioned and the Cu samples could not be processed. The following conclusions, however, were reported:

(1) The main result from the experiment was that the space-processed samples contained a more homogeneous distribution of particles than the 1-g processed samples. However, even low-gravity processing did not achieve a completely homogeneous distribution, indicating the presence of gravity-independent, particle rearrangement mechanisms.

(2) The macro-hardness of the space samples (Samples 1, 2, and 4) was more uniform than the 1-g samples.

(3) From elementary fracture analysis, the adhesion between the SiC particles and Al was improved by low-gravity melt processing (compared to initial powder metallurgically prepared samples). This improved adhesion was especially apparent in samples with coarse particles.

(4) The difference in particle distribution between the flight and ground-based samples were attributed to (a) sedimentation, (b) fluid flow from thermal convection, and particularly (c) agglomeration tendency of the dispersed particles. It was also reported that the mutual wetting behavior of the matrix material and dispersed phase was an important factor in particle distribution (see also Deruyterre, TEXUS 7).

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Ternary Systems, Reinforced Materials, Powder Metallurgy, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Particle Agglomeration, Particle Coalescence, Solid/Liquid Dispersion, Solid/Liquid Interface, Solid/Liquid/Gas Dispersion, Homogeneous Dispersion, Stability of Dispersions, Stability of Suspensions, Suspension of Particles, Sedimentation, Interface Physics, Interface Phenomena, Inclusion and/or Rejection of Particles, Wetting, Particle Wetting, Surface Energy, Isothermal Processing, Thermal Soak, Thermal Convection, Hardness, Tensile Strength, Material Strength, Furnace Malfunction

Number of Samples: 6 (although 12 were intended)
Sample Materials: Samples actually processed: aluminum matrix samples containing alumina or silicon carbide particles; samples which were not processed as planned: six copper matrix samples containing alumina, tungsten, molybdenum or silicon carbide particles
(Al*O*, Al*Si*C*, Al*, Cu*Al*O*, Cu*, Cu*W*, Cu*Mo*, Cu*Si*C*)
Container Materials: Graphite crucibles contained in tantalum cartridge
(C*, Ta*)

Experiment/Material Applications:
Light aluminum-based composites with particulate ceramic reinforcements were selected for fundamental and technical reasons. The systems Al-Al_2O_3 and Al-SiC are quite well understood from an interaction point of view. Also the wetting behavior of the ceramic phase by the liquid metal is well described.

These materials offered potential in many technical applications, for which lightweight and high specific properties are required. (Reference (8))

References/Applicable Publications:

(1) Froyen, L. and Deruyttere, A.: Melting and Solidification of Metal Matrix Composites Under Microgravity. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 69-78.

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication (acceleration measurements on Spacelab 1)

(3) Froyen, L. and Deruyttere, A.: Metallic Composite Materials and Microgravity. In Proceedings of the 4th European Symposium on Materials Science Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, p. 36. (preflight)

(4) Deruyttere, A., Froyen, L., and De Bondt, S.: Melting and Solidification of Metallic Composites in Space. Adv. Space Res., Vol. 6, No. 5, pp. 101-110, 1986. (post-flight results from several microgravity experiments)

(5) Whittmann, K: The Isothermal Heating Facility. In ESA 5th European Symposium on Materials Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 49-54. (IHF facility)

(6) Froyen, L. and Deruyttere, A.: The Behavior of Dispersed Particles in Molten Metal Matrix Composites. 21 Int. Scientific Meeting in Space, Rome, March 25-26, 1981, pp. 133-142. (preflight)

(7) Input received from Principal Investigator A. Deruyttere, June 1988.

(8) Input received from Principal Investigator L. Froyen, August 1993.

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Co-Investigator(s): None
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Experiment Origin: Belgium
Mission: TEXUS 9
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Multipurpose Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Particle Composites - Dispersion Alloys

This TEXUS 9 experiment was the fifth in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab SL-3 (Chapter 4), TEXUS 6, TEXUS 7, Spacelab 1). The specific objective of the experiment was to determine the role of a moving solidification front on the distribution of particles in the solidified composite. This objective was to be achieved by studying identical samples subjected to different thermal conditions.

Prior to launch, four samples were prepared: Samples 1 and 3 were a powder metallurgical Cu-Mo composite (0.5 vol.% Mo, 0-45 microns) and Samples 2 and 4 were a powder metallurgical CuSiO_2 composite (1 vol.% SiO_2, 0-32 microns). Reportedly, the Mo particles are well-wetted by the copper melt and the SiO_2 particles are poorly-wetted by the copper melt. Prior to enclosure in poorly-wetted graphite crucibles, the four samples were treated in a reducing H_2 atmosphere (1000 ^{o}C for 8 hours). All four crucibles were then sealed in a single TZM cartridge.

The cartridge was placed in the TEXUS Experiment Module TEM 01-1 Multipurpose Furnace such that Samples 3 and 4 were in the gradient section of the apparatus. "The gradient side of the cartridge was connected to a heat sink by using a copper cooling block. The heat sink contained... [paraffin]. During cooling, the...[paraffin] underwent a solid...[to] liquid transformation, extracting extra heat." (5) The samples experienced the following thermal gradients: Sample 1- 3.8 ^{o}C/mm; Sample 2- 2.2 ^{o}C/mm; Sample 3- 6.4 ^{o}C/mm, and Sample 4- 11.4 ^{o}C/mm. Reportedly the experiment fulfilled the requested conditions. It appears that melting and solidification of the samples occurred during the low-gravity period of the mission.

Post-flight analysis of the low-gravity samples indicated that the redistribution of particles was less pronounced than that found in samples similarly produced on Earth. In the isothermal flight Cu-SiO_2 sample, the particles moved from the lower part of the sample to the top and exuded from the matrix. However, in flight sample 4 (highest gradient), the Mo particles retained a good distribution throughout the copper matrix. In the gradient Cu-SiO_2 flight samples, a portion of the particles remained in the matrix while some were exuded from the copper.

It was reported that there was no significant difference between the ground-based samples processed (1) isothermally or (2) in the gradient portion of the furnace. Examination of the 1-g processed Cu-Mo samples indicated a complete redistribution of the Mo particles: they remained in the Cu matrix but moved toward the side surfaces. In the ground-based SiO_2 samples, most of the particles moved from their initial position (lower portion of the sample) and exuded from the top of the sample.

It was concluded that low-gravity levels with gradient conditions appeared to favor a stable dispersion of well-wetted Mo particles within a Cu matrix. The separation of poorly-wetted SiO_2 particles from the copper could be explained by interfacial energy considerations.

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Powder Metallurgy, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Stability of Suspensions, Suspension of Particles, Sedimentation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Wetting, Particle Wetting, Surface Energy, Interfacial Energy, Isothermal Processing, Thermal Gradient

Number of Samples: four
Sample Materials: Samples 1 & 3: powder metallurgical copper-molybdenum composite (0.5 vol.% Mo, 0-45 microns); Samples 2 & 4: powder metallurgical copper-silica composite (1 vol.% SiO_2, 0-32 microns)
(Cu*Mo*, Cu*Si*O*)
Container Material: graphite crucible within a TZM cartridge
(C*)

Experiment/Material Applications:
See Deruyttere, TEXUS 6, TEXUS 7.

References/Applicable Publications:
(1) Froyen, L.: De Invloed van Het Smelten Op Metaalkomposieten, Doctoral Thesis, K. U. Leuven, 1984.

(2) Deruyttere, A., Froyen, L., and De Bondt, S.: Melting and Solidification of Metallic Composites in Space. Adv. Space Research, Vol. 6, No. 5, 1986, pp. 101-110.

(3) Input received from Principal Investigator A. Deruyttere, June 1988.

(4) Melting and Solidification of Metallic Composites. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 300-301. (post-flight)

(5) Input received from Principal Investigator L. Froyen, August 1993.

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Principal Investigator(s): Deruyttere, A. (1), Froyen, L. (2)
Co-Investigator(s): None
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Experiment Origin: Belgium
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metallic Particle Composites - Dispersion Alloys (WL-IHF 08)

This Spacelab D1 experiment was the sixth in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab SL-3 (Chapter 4), TEXUS 6, TEXUS 7, Spacelab 1, TEXUS 9). The specific objective of the experiment was to further understand and control metallurgical production of composite materials.

The six sample compositions selected for evaluation were to have been processed on the earlier Spacelab 1 flight. (A malfunction during Spacelab 1 prevented examination of copper matrix samples (see Deruyttere, Spacelab 1).) Prior to the Spacelab D1 flight, the samples were prepared in a fully dense, reduced form (H_2 atmosphere, 850 ^{o}C): (Sample 1) an oxide-dispersion-strengthened (ODS) alloy, Cu-Al matrix with approximately 3 wt.% Al_2O_3 particles (0.1-0.5 microns, rounded), (Sample 2) a Cu matrix with 0.5 wt.% W particles (20-45 microns spherical), (Sample 3) a Cu matrix with 0.5 wt.% Mo particles (20-45 microns, spherical), (Sample 4) a Cu matrix with 2 wt.% Mo particles (250-355 microns, spherical), (Sample 5) a Cu matrix with 1 wt.% SiC particles (0-15 microns, spherical), and (Sample 6) a Cu matrix with 0.1 wt.% SiC particles (0-15 microns, spherical). The samples were contained within a single graphite crucible and configured within the Spacelab D1 Isothermal Heating Facility (IHF). Reportedly, "The design of the graphite crucible... was the same as for the SPACELAB-1 experiments." (6, p. 317)

During the low-gravity mission, the samples were (1) heated to 1250 ^{o}C, (2) held at this temperature for 470 minutes, and then (3) solidified. Unexpectedly "...rather high thermal gradients led to a solidification from the sample ends towards the centre."

(6, p. 321) This resulted in shrinkage holes located at the center of the sample.

Post-flight, Spacelab-D1 and 1-g processed materials were compared. The following conclusions were presented:

(1) The Cu-W and Cu-Mo flight samples contained a rather inhomogeneous suspension. This result was attributed to the presence of gas bubbles within the melt. The bubbles were formed by the decomposition of MoO_3 and WO_3 layers. (These oxide layers become volatile above approximately 800 ^{o}C.) The oxidation of the particles probably occurred during the sample preparation stage. Also, other particle redistribution mechanisms (e.g., agglomerations) were detected.

(2) In the Spacelab Cu-SiC samples, the expected SiC decomposition occurred. However, the Cu(Si)-C suspension was not stable. The copper matrix contained Si in solid solution but no dispersed graphite. The SiC particles were almost completely exuded from the matrix.

(3) The Cu-Al_2O_3 flight sample contained a stable dispersion and remained homogeneous during processing. A limited growth of Al_2O_3 particles was observed. The flight sample was significantly harder than the 1-g processed sample, which contained pronounced particle agglomeration.

It was reported that these results "...demonstrated the practability <sic> of the recasting (e.g., during welding) of ODS-alloys in a... [microgravity]... environment." (6, p. 321)

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Model Materials, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Particle Agglomeration, Particle Growth, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Stability of Suspensions, Suspension of Particles, Sedimentation, Bubbles, Interface Physics, Inclusion and/or Rejection of Particles, Surface Energy, Interfacial Energy, Oxide Layer, Isothermal Processing, Thermal Soak, Thermal Gradient, Oxidation, Hardness, Sample Shrinkage, Thermal Environment More Extreme Than Expected, Processing Difficulties

Number of Samples: six
Sample Materials: copper matrix samples with alumina, tungsten, molybdenum, or silicon-carbide dispersions
(Cu*Al*O*, Cu*W*, Cu*Mo*, Cu*Si*C*)
Container Material: graphite crucible
(C*)

Experiment/Material Applications:
The sample materials selected for this experiment have the following technological applications:

(1) Cu-Al_2O_3 is (a) a good electrical conductor with excellent high temperature strength and (b) a good model system for other ODS alloys.

(2) Cu-W (with high particle content) is an electrical contact material with excellent abrasive properties.

(3) Cu-Mo (with high particle content) is an electrical contact material with excellent abrasive properties.

(4) Cu-SiC is an electrical contact material.

See also Deruyttere, TEXUS 6.

References/Applicable Publications:
(1) Froyen, L. and Deruyttere, A.: Melting and Solidification of Metallic Composite Materials. In Naturwissenschaften, 73. Jahrgang Heft 7, July 1986, pp. 384-386. (post-flight)

(2) Input received from Principal Investigator A. Deruyttere, June 1988.

(3) Deruyttere, A., Froyen, L., and De Bondt, S.: Melting and Solidification of Metallic Composites in Space. Adv. Space Res., Vol. 6, No. 5, 1986, pp. 101-110. (post-flight)

(4) Froyen, L. and Deruyttere, A.: Melting and Solidification of Metallic Composite Materials. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 78-79. (post-flight)

(5) Deruyttere, A. and Froyen, L.: Melting and Solidification of Metallic Composites. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 137. (preflight)

(6) Froyen, L. and Deruyttere, A.: Melting and Solidification of Copper Matrix Composite Materials. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 315-321. (post-flight)

(7) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements)

(8) Input received from Principal Investigator L. Froyen, August 1993.

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Co-Investigator(s): None
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Experiment Origin: Belgium
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation experiment module containing the Gradient Furnace Assembly (GFA)
Builder of Processing Facility: Swedish Space Corporation (SSC)/ACR, Solna, Sweden

Experiment:
Metallic Particle Composites

This TEXUS 14a sounding rocket experiment was the seventh in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere Skylab 3, TEXUS 6, TEXUS 7, Spacelab 1, TEXUS 9, Spacelab D1). The specific objective of the experiment was to further understand the sedimentation and skeletal formation processes in melted and solidified composites.

During the mission, two furnaces within the Swedish Space Corporation experiment module were to melt and resolidify two Al-SiC samples (0.4 vol.% SiC, 0-3 microns). The samples (designated as sample A and sample B) were to be processed with identical heating cycles but different solidification rates. This method of processing was employed to investigate the influence of melting and solidification on particle redistribution.

Reportedly, the TEXUS 14a experiment was not processed in the expected low-gravity environment. Due to an unexpected "wobbling motion" of the TEXUS rocket, uncontrollable accelerations were produced on the vehicle and the desired low gravity of 10^{-4} g was not attained.

Principal Investigator L. Froyen reported that the samples were not analyzed. The experiment was reflown on TEXUS 14b (see Deruyttere, TEXUS 14b).

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Melt and Solidification, Casting, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Sedimentation, Interface Physics, Inclusion and/or Rejection of Particles, Surface Energy, Interfacial Energy, Rocket Motion, Acceleration Effects, Coated Surfaces

Number of Samples: two
Sample Materials: Al-0.4 vol.% SiC, 0-3 micron particle size (Al*Si*C*)
Container Material: thin stainless steel cartridge, internally coated with a thin graphite layer
(C*)

Experiment/Material Applications:
See Deruyttere, TEXUS 6 and Spacelab 1.

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR Abschlussbericht 1988, pp. 53-55. (in German; postflight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR Abschlussbericht 1988, pp. 60-61. (gradient furnace assembly)

(3) Input received from Principal Investigator L. Froyen, August 1993.

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Principal Investigator(s): Deruyttere, A. (1), Froyen, L. (2),
Co-Investigator(s): None
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Experiment Origin: Belgium
Mission: TEXUS 14b
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation experiment module containing the Gradient Furnace (GFA)
Builder of Processing Facility: Swedish Space Corporation (SSC)/ACR, Solna, Sweden

Experiment:
Metal Matrix Composites

This TEXUS 14b experiment was the eighth in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab SL-3, TEXUS 6, TEXUS 7, Spacelab 1, TEXUS 9, Spacelab D1, and TEXUS 14a). The specific objective of the experiment was to further understand the sedimentation and skeletal formation processes in melted and solidified composites.

During the mission, two furnaces within the Swedish Space Corporation experiment module were used to melt and resolidify two Al-SiC samples (0.4 vol.% SiC, 0-3 microns). The samples (designated as Sample A and Sample B) were processed with identical heating cycles (heating to 750 ^{o}C (T_m + 90)) but then cooled at different rates to establish different solidification velocities. This method of processing was used to investigate the influence of melting and solidification on particle redistribution. Reportedly, "The redistribution of the particles during melting could be studied in the fast cooled sample...[in] holder A... where the particle distribution after melting was conserved." (4, p. 302) "With the sample in holder B, it is possible to obtain the influence of a slowly moving solidification front on the dispersed particles (0.4μ/s: the slowest rate possible within the 5 min cooling time in reduced gravity)." (1, p. 70)

Reference experiments were performed on Earth using a gradient furnace identical to the flight furnace. Calculations which simulated the directional solidification process (taking into account the difference in thermal conductivity between the particle and matrix materials) were performed to aid in the analysis of the samples (see Reference (2) for details).

Post-flight examination of the thermal data and sample dendritic arm spacing revealed a solidification rate of (1) 8 microns/s for Sample A and (2) 0.4 microns/s for Sample B. The flight thermocouple readings indicated that the samples experienced significantly longer solidification times than (1) observed during the 1-g reference tests and (2) predicted by the computer simulations. Radiographic examination of both the low-gravity and 1-g reference samples revealed the presence of unexpected, large holes in the material. "No such hole formation had ever been observed during the [other] extensive ground tests." (4, p. 302) These holes were attributed to defects and inhomogeneities (adsorbed hydroxides on the Al-powder) introduced during preflight sample fabrication. This was indicated by the following:

(1) No such large holes were present in the samples before processing.

(2) Aluminum had been pressed into the plug during processing because of the expansion of gas trapped in the samples. <Note: It appears that the "plug" acted as a cap on one end of the sample holder.>

(3) Some holes were covered with SiC particles; these holes were more spherically shaped than those holes around internal oxide surfaces.

The following effects of the gas hole formation were reported:

(1) A different particle distribution resulted than what was expected. This distribution was attributed to different melt flows around the holes.

(2) A slower heat transfer rate resulted. This slower rate was the main reason the predicted and observed solidification times differed.

These effects reduced the use of Sample A as a reference to Sample B and impeded the quantitative analysis of the experiment. However, qualitative conclusions were reported: "...contrary to ground test and as predicted by an expulsion model...[see Reference (2)]..., macroscopic interaction of the dispersed particles with the solidification front has been shown. Also microscopic redistribution (by dendrites) was observed." (1, p. 73)

It was reported that "...the main results of the experiment TEXUS 14B are the development of a new sample-crucible design with a highly improved thermal control [see References (1) and (2)], and the qualitative confirmation of expectations based on a new particle-solidification front interaction model. Its merits are

mainly the formulation of an expulsion force that can be calculated and the deduction of the importance of gravity. The difference in heat conductivity between a particle and the matrix is most important." (1, p. 74)

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Powder Metallurgy, Melt and Solidification, Directional Solidification, Thermal Gradient, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Voids, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Sedimentation, Interface Physics, Inclusion and/or Rejection of Particles, Surface Energy, Interfacial Energy, Solidification Front Physics, Heat Transfer, Solidification Rate, Dendritic Arm Spacing, Dendrites, Coated Surfaces, Defects

Number of Samples: two
Sample Materials: Al-0.4 vol.% SiC, 0-3 micron particle size (Al*Si*C*)
Container Material: thin stainless steel cartridge internally coated with a thin graphite layer
(C*)

Experiment/Material Applications:
See Deruyttere, TEXUS 6 and Spacelab 1.

References/Applicable Publications:
(1) Deruyttere, A., Froyen, L., and De Bondt, S.: Metal Matrix Composites. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 70-74. (post-flight)

(2) Deruyttere, A., Froyen, L., and De Bondt, S.: Melting and Solidification of Metallic Composites in Space. Adv. Space Research, Vol. 6, No. 5, pp. 101-110 (1986).

(3) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 60-61. (gradient furnace assembly)

(4) Metal Matrix Composites. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 302-303. (post-flight)

(5) Input received from Principal Investigator A. Deruyttere, June 1988.

(6) Input received from Principal Investigator L. Froyen, August 1993.

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Experiment Origin: Belgium
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metal Matrix Composites

This TEXUS 16 experiment was the ninth in a series of investigations designed by Deruyttere et al. to study the low-gravity melting and solidification of metallic composite materials (see Deruyttere, Skylab 3, TEXUS 6, TEXUS 7, Spacelab 1, TEXUS 9, Spacelab D1, TEXUS 14, and TEXUS 14b).

The specific TEXUS 16 experiment goals and equipment setup were not detailed in the available publications.

Reportedly, the experiment did not proceed as planned during the mission because of a rocket system malfunction. Shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached, and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all of the experiment modules including the TEM 01-2 module.

The experiment was reflown on TEXUS 20.

<Note: The results of Deruyttere's experiment on TEXUS 20 is beyond the scope of this publication. These results will be reported in later versions of this technical memorandum.>

Key Words: Composites with Solid Particles, Melt and Solidification, Metallic Matrix, Particle Dispersion, Particle Distribution, Solid/Liquid Dispersion, Solid/Liquid Interface, Rocket Motion, Acceleration Effects, Rocket Failure, Payload Survivability

Number of Samples: unknown
Sample Materials: unknown
Container Materials: unknown

Experiment/Material Applications:
See Deruyttere, TEXUS 6.

References/Applicable Publications:
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 109-111. (in German; post-flight)

(2) Input received from L. Froyen (Katholieke Universiteit), August 1993.

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Co-Investigator(s): Takahashi, S. (2)
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Experiment Origin: Japan
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt. Date: September 1973 (month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF), located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Whisker Reinforced Composites (M561)

This Skylab SL-3 experiment was the first in a series of investigations designed by Kawada et al. to study the low-gravity solidification of whisker-reinforced composite materials. The specific objective of the experiment was to produce void-free silver samples which had been reinforced with oriented silicon carbide whiskers.

Prior to the flight, three Ag-SiC samples were prepared: (1) Ag-2 vol.% SiC, (2) Ag-5 vol.% SiC, and (3) Ag-10 vol.% SiC. The 8 mm diameter, 35 mm long samples were prepared by (1) mixing Ag powder (less than 0.5 microns in diameter) with SiC whiskers (on average 0.1 micron diameter, 10 microns long), (2) compacting the powder mixtures, (3) sintering the powder mixtures in a HP_2 atmosphere at 900 ^{o}C, and (4) hot pressing the sintered materials. Each sample was placed in a graphite sheath surrounded by a silica tube. A coiled spring with a piston rod was placed at one end to pressurize the sample and crush the entrapped voids. The entire sample/sheath/piston assembly was sealed within the Skylab Multipurpose Electric Furnace System (MEFS).

During the experiment, the furnace was (1) heated to 1010 ^{o}C and (2) held at this temperature for 4 hours (such that the samples attained a completely molten state). The furnace was then switched off and the samples were allowed to cool.

Reference samples were prepared on Earth for comparison to the Skylab specimens. During these 1-g experiments, "...the longitudinal axis of [the] sample and [the] sample ampoule were held vertical to the ground, and the piston rod was kept downward to the sample." (2, p. 453) Reportedly, the 1-g samples remained molten for approximately 2 hours.

Post-flight, the following characteristics of the 1-g and space-processed samples were investigated: (1) macrostructure, (2) microstructure, (3) whisker distribution, (4) x-ray structure, (5) microhardness, (6) bend-test strength, and (7) fracture surface. From these examinations, the following conclusions were reported:

(1) The density ratio (measured density/theoretical density) of both the 1-g and low-gravity processed samples was increased because of the employed spring/piston ampoules.

(2) Crystal grain sizes of the 1-g and space-processed samples at the spring/piston end were about one-tenth those at the opposite end because of (a) heat conduction through the graphite piston rod and (b) thermal characteristics of the MEFS.

(3) Separation of the whiskers and Ag in the low-gravity samples was not observed, despite the large density difference between Ag and SiC. However, separation, on a microscopic and macroscopic scale, was observed in the 1-g processed samples.

(4) The microhardness values and whisker distribution density from the low-gravity samples were uniform over the entire sample. "The whiskers of the 1-g sample were distributed richly at the spring end compared to the other end, and the values of hardness were scattered widely with their measured positions." (2, p. 457)

(5) X-ray analysis indicated a preferred grain orientation in the space-processed samples. This orientation was in the large grained area of the sample (opposite from the spring/piston end of the sample). The 1-g processed samples did not indicate such an orientation.

(6) The bend tests indicated a ductile behavior for the low-gravity processed samples while the 1-g samples exhibited a brittle behavior.

Reportedly, these results "...indicate clearly that the low-g samples were devoid of [the] influence of floating due to the density difference and thermal convection: accordingly, the low-gravity environment is a preferable place where the high-quality composite materials can be well processed." (2, p. 457)

Key Words: Composites with Solid Particles, Binary Systems, Reinforced Materials, Dispersion Alloys, Metallic Matrix, Powder Metallurgy, Melt and Solidification, Whiskers, Thermal Convection, Heat Conduction, Buoyancy Effects, Density Difference, Sedimentation, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Thermal Soak, Sample Microstructure, Grain Size, Hardness, Material Strength, Piston System, Separation of Components, Voids, Passive Cooling

Number of Samples: three
Sample Materials: (1) Ag-2 vol.% SiC whiskers, (2) Ag-5 vol.% SiC whiskers, and (3) Ag-10 vol.% SiC whiskers
(Ag*Si*C*)
Container Materials: graphite sheath contained in silica tube which was sealed in a stainless steel cartridge
(C*, Si*O*)

Experiment/Material Applications:
Composite materials, composed of high-strength whiskers within a metal matrix, have high-strength applications.

The Ag-SiC material was selected for these experiments because (1) it could be processed within the temperature limits of the MEFS, (2) its constituents, Ag and SiC, have large density differences, (3) there is little or no chemical reaction between Ag and SiC, and (4) the Ag and SiC particle size and shape can be well controlled.

References/Applicable Publications:
(1) Kawada, T., Takahashi, S., Yoshida, S., Ozawa, E., and Yoda, R.: Preparation of Silicon Carbide Whisker Reinforced Silver Composite Material in a Weightless Environment-Skylab Experiment M561. In Proceedings of the Third Space Processing Symposium Skylab Results, Vol. 1, April 30-May 1, 1974, Marshall Space Flight Center, Huntsville, Alabama, June 1974, pp. 203-234. (post-flight SL-3 only)

(2) Takahashi, S.: Preparation of SiC Whisker Reinforced Silver Composite Material on Skylab. AIAA Journal, Vol. 16, No. 5, May 1978, pp. 452-457. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(4) Experiment M561- Whisker-Reinforced Composites. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-69 - 5-72. (post-flight)

(5) Naumann, R. J. and Herring, H. W.: Experiment M561, Silicon Carbide Whisker Reinforced Silver Composite Material. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, p. 70. (post-flight)

(6) M518 Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(7) Experiment M561- Whisker-Reinforced Composites. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-69 - 5-72. (post-flight)

(8) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report- Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing Facility)

(9) Whisker-Reinforced Composites (M561). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-50 - 12-51. (post-flight; very short summary)

(10) Naumann, R. J. and Mason, D.: Whisker Reinforced Composites. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 30. (post-flight)

(11) Input received from Co-Investigator S. Takahashi, August 1993.

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Experiment Origin: Japan
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: December 1973 (month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels, located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Whisker Reinforced Composites (M561)

This Skylab SL-4 experiment was the second in a series of investigations designed by Kawada et al. to study the low-gravity solidification of whisker reinforced composite materials (see Kawada, Skylab SL-3).

No publication which specifically discusses the objectives, experimental setup, or results from this experiment could be located. The Co-Investigator briefly reported that the temperature attained by the employed furnace was not high enough to melt the SL-4 samples. Subsequently, no results were reported.

Please refer to Kawada, Skylab SL-3 for further information.

Key Words: Composites with Solid Particles, Binary Systems, Reinforced Materials, Dispersion Alloys, Metallic Matrix, Melt and Solidification, Whiskers, Buoyancy Effects, Density Difference, Sedimentation, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Sample Microstructure, Material Strength, Incomplete Sample Processing

Number of Samples: three
Sample Materials: (1) Ag-2 vol.% SiC whiskers, (2) Ag-5 vol.% SiC whiskers, and (3) Ag-10 vol.% SiC whiskers
(Ag*Si*C*)
Container Materials: Unknown, possibly graphite sheath contained in silica tube which was sealed in a stainless steel cartridge.
(C*, Si*O*)

Experiment/Material Applications:
See Kawada, Skylab SL-3.

References/Applicable Publications:
<Note: The following references discuss the Skylab Whisker Reinforced Composites Experiments in general as well as detailing results from the earlier SL-3 mission. They do not detail results from the SL-4 mission, (except Reference (10)).>

(1) Kawada, T., Takahashi, S., Yoshida, S., Ozawa, E., and Yoda, R.: Preparation of Silicon Carbide Whisker Reinforced Silver Composite Material in a Weightless Environment-Skylab Experiment M561. In Proceedings of the Third Space Processing Symposium Skylab Results, Vol. 1, April 30-May 1, 1974, Marshall Space Flight Center, Huntsville, Alabama, June 1974, pp. 203-234. (post-flight; SL-3 only)

(2) Takahashi, S.: Preparation of SiC Whisker Reinforced Silver Composite Material on Skylab. AIAA Journal, Vol. 16, No. 5, May 1978, pp. 452-457. (post-flight; SL-3 only)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements)

(4) Experiment M561- Whisker-Reinforced Composites. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-69 - 5-72. (post-flight)

(5) Naumann, R. J. and Herring, H. W.: Experiment M561, Silicon Carbide Whisker Reinforced Silver Composite Material. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, p. 70. (post-flight)

(6) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(7) Experiment M561- Whisker-Reinforced Composites. In MSFC Corollary Experiment Systems Evaluation, NASA TM X-64820, September 1974, pp. 5-69 - 5-72. (post-flight)

(8) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report - Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(9) Whisker Reinforced Composites (M561). In MSFC Skylab Mission Report - Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-50 - 12-51. (post-flight; very short summary)

(10) Input received from Co-Investigator S. Takahashi, August 1993.

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Co-Investigator(s): None
Affiliation(s): (1,2) During SPAR 1: The Aerospace Corporation, Los Angeles, California; (1) Currently: L. Raymond and Associates, Newport Beach, California; (2) Currently: Deceased

Experiment Origin: USA
Mission: SPAR 1
Launch Date/Expt Date: December 1975
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: General Purpose Rocket Furnace
Builder of Processing Facility: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Casting Dispersion - Strengthened Composites (74-34)

This SPAR 1 sounding rocket experiment was the first in a series of investigations designed by Raymond et al. to study the low-gravity casting of dispersion-strengthened metal matrix composite materials. The overall goal of the investigative series was to determine the feasibility of, and establish techniques for, reduced gravity casting of new dispersion-strengthened titanium matrix composites. Magnesium (rather than titanium) was used to model the technique because its lower melting point was within the temperature limitations of the employed SPAR furnace. (Both magnesium and titanium have identical thermodynamic behavior relative to oxidation.)

The specific objective of the SPAR 1 experiment was to evaluate the retention of a hot pressed dispersion of heavy thoria particles in low-density magnesium after low-gravity, short-duration melting and solidification.

Prior to the mission, a single, pre-mixed hot-pressed Mg-10.5 wt.% (2 vol.%) ThO_2 sample was configured in an argon-filled "POCO" graphite crucible and placed within the SPAR General Purpose Rocket Furnace. It was reported that during the mission, (a) the sample was heated at a rate of 10 ^{o}C/s and (b) the sample's temperature was maintained above the Mg melting point (651 ^{o}C) for the duration of the low-gravity period (230 seconds). (The sample was held at greater than or equal to 800 ^{o}C for at least 190 seconds during the low-gravity period.) Additional samples were similarly processed on Earth for comparison.

Reportedly, "...the flight furnace did not follow the programmed cooldown rate after 340 seconds because of a malfunction of the gas injection system. However,... [it was believed]... that the sample had already solidified completely by that time." (1, p. VI-6)

Post-flight examination of the flight sample revealed gas voids located next to the crucible wall and "...dispersion-depleted areas, relatively large in size..." which were roughly round in shape. (1, p. VI-11) A dispersion-depleted area was also located around the periphery of the flight sample. The ground-processed samples contained voids internal to the sample material and layered sedimentation of the dispersoids due to gravity. One ground-based sample contained voids located on and near the top of the ingot. All samples (flight and ground processed) showed sedimentation or clusters of dispersoids in two layers. However, the layering due to gravity was predominant in the ground samples. (A detailed discussion of the sample microstructure is contained in Reference (1).)

A summary of the possible mechanisms acting on the samples was reported as follows:

"The internally sound ingot cast under low-gravity conditions, in contrast to the one-gravity samples, is a beneficial manifestation of low-gravity effects in preventing sedimentation in the heavy ThO_2 particles in the liquid state. The protective gas argon moved to the container walls and the non-wetting melt moved toward the center of the crucible." (5)

"The presence of rounded regions that are depleted in dispersion can be attributed to thermoacoustic waves induced by rapid heating, coupled with the mechanism described in item 1 and accompanied by void-filling action of the low viscosity liquid.

"Overheating and heating with gas beneath the melt under the influence of gravity will result in extremely unsound ingots with internal voids and shrinkage cavities....

"Layered and nonuniform sedimentation of dispersoids in all ingots examined represents a deviation from the ideal Stokes law and is not unexpected with the present experiment design. It is necessary, however, to speculate on the possibility of the flight sample's excursion at the 10^{-2} to 10^{-3} gravity levels during initial melting to explain the observed layered sedimentation.

"The presence of the dispersion-depleted periphery of the flight-processed ingot cannot be explained by the particle rejection mechanism because of the estimated high rate of solidification. However, the mechanism of preferential gas motion at low gravity

coupled with surface tension induced liquid motion and Mg vapor condensation can be used to explain this particular microstructural feature." (1, p. VI-31)

These results led to the following recommendations for the SPAR 2 experiment on cast dispersion-strengthened composites (with the objective of attaining the liquid state while only at 10^{-4}g to prevent sedimentation due to gravity effects):

(1) Preheat the sample cartridges below the melting point of Mg to a temperature of 400 ^{o}C to 500 ^{o}C prior to launch.

(2) Apply a heating rate of less than 4 ^{o}C/s and prevent sample melting prior to the achievement of a gravity level less than 10^{-4} g.

(3) Establish a sample soak temperature in the range of 800 ^{o}C to 850 ^{o}C which may be as short as 180 seconds.

(4) Reduce the thermal lag between the sample and furnace and apply a quench rate which is as rapid as possible in order to solidify before re-entry an attainment of 10^{-2} g to 10^{-3} g gravity levels while in the liquid state.

(See Raymond, SPAR 2, for more complete information on the SPAR 2 experimental objectives and results.)

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Model Materials, Melt and Solidification, Casting, Metallic Matrix, Dispersion Strengthening, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Interface Physics, Inclusion and/or Rejection of Particles, Voids, Cavity, Wetting, Capillary Flow, Surface Tension, Condensation, Sedimentation, Viscosity, Thermoacoustic Effects, Crucible Effects, Non-Wetting of Container, Thermal Environment More Extreme Than Predicted, Thermal Soak, Quench Process, Solidification Rate, Sample Microstructure, Acceleration Effects, Sample Shrinkage, Material Strength, Hardware Malfunction

Number of Samples: one
Sample Materials: Mg-12.3 wt.% ThO_2 particles (Mg*Th*O*)
Container Materials: "POCO" graphite

Experiment/Material Applications:
Gravity-induced sedimentation and buoyancy effects restrict the production of dispersion-strengthened metal matrix composites of titanium or magnesium on Earth. If the cast composites were liquefied in a near-zero gravity environment, the resulting composite materials may exhibit increased strength at high temperatures.

References/Applicable Publications:
(1) Raymond, L. and Ang, C. Y.: Casting Dispersion-Strengthened Composites at Zero Gravity. In Space Processing Applications Rocket Project, SPAR I Final Report, NASA TM X-3458, pp. VI-1 - VI-34, December 1976. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements SPAR 1-4)

(3) Naumann, R. J. (editor): Casting Dispersion-Strengthened Composites at Zero Gravity. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, NASA TM-78217, January 1979, pp. 7-8. (post-flight)

(4) Raymond, L. and Ang, C. Y.: Casting Dispersion-Strengthened Composites at Zero Gravity. In Space Processing Applications Rocket Project SPAR II - Final Report, NASA TM-78125, pp. VII-1 - VII-51, November 1977. (post-flight)

(5) Input received from Principal Investigator L. Raymond, August 1993.

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Co-Investigator(s): None
Affiliation(s): (1,2) During SPAR 2: The Aerospace Corporation, Los Angeles, California; (1) Currently: L. Raymond and Associates, Newport Beach, California; (2) Currently: Deceased

Experiment Origin: USA
Mission: SPAR 2
Launch Date/Expt Date: May 1976
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: FWD (Forward) and Aft General Purpose Rocket Furnaces
Builder of Processing Facility: Unclear, possibly The National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Casting Dispersion - Strengthened Composites (74-34)

This SPAR 2 sounding rocket experiment was the second in a series of investigations designed by Raymond et al. to study the low-gravity casting of dispersion-strengthened metal matrix composite materials (see Raymond, SPAR 1). The overall goal of the investigative series was to determine the feasibility of, and establish techniques for, reduced gravity casting of new dispersion-strengthened titanium matrix composites. Magnesium (rather than titanium) was used to model the technique because its lower melting point was within the temperature limitations of the employed SPAR furnace. (Both magnesium and titanium have identical thermodynamic behavior relative to oxidation.)

The specific objectives of the SPAR 1 and SPAR 2 experiments were (1) to observe the stability of pre-existing dispersed ThO_2 particles in the molten Mg matrix at low-g [SPAR 1]; (2) to determine if an in situ reaction-precipitation ThO_2 dispersion could be established (via the gettering mechanism) under low-g conditions [SPAR 2]; and (3) to determine low-g effects on the resulting dispersion-strengthened Mg matrix by comparing flight and ground-based samples processed under identical thermal conditions.

During the earlier SPAR 1 mission, a premixed powder sample of Mg containing already formed ThO_2 particles was melted and solidified retaining a dispersion-strengthened metal matrix composite under low-gravity conditions. During this SPAR 2 mission, two samples comprised of Mg-9.2 wt.% Th-3.2 wt.% MgO were stoichiometrically selected such that ThO_2 particles were formed in the liquid state by the following reaction:

$2\ MgO + Th \rightarrow ThO_2 + 2Mg$

and were retained during solidification at low-g.

This reaction allowed the in situ production of 2 vol.% ThO_2 particles. Thus, the composite was produced by taking advantage of the ability of Th to getter oxygen from MgO and precipitate out as ThO_2 particles within the Mg matrix.

During the SPAR 2 mission, the forward and aft portions of the General Purpose Rocket Furnace (GPRF) were used to process the two samples. Each sample was contained within an argon-filled "POCO" graphite crucible. The Ar was included because (1) the high vapor pressure of Mg would be detrimental in terms of metal evaporation and vapor condensation, (2) the oxidation of Mg during preflight sample preparation had to be minimized (the samples were prepared in a glovebox under an Ar atmosphere), and (3) any practical casting of Mg is performed in a flux which precludes the use of a vacuum.

The experimental procedures for both SPAR 2 samples were as follows: (1) pre-launch thermal soak at approximately 500 ^{o}C, (2) furnace heat-up at a rate of less than 10 ^{o}C/sec, (3) thermal soak in the range of 800 ^{o}C to 950 ^{o}C under low-gravity conditions, and (4) He quench at a rate greater than or equal to 10 ^{o}C/sec. The intended low-gravity soak time for the forward furnace sample was approximately 60 seconds and the intended low-gravity soak time for the aft furnace sample was greater than or equal to 180 seconds.

Reportedly, "The short-soak flight test in the Forward Furnace appeared to have failed, because the interpolated cartridge temperature had barely reached 800 ^{o}C when quenching was initiated." (1, p. VII-26). Further, the presoak times of the two flight samples, as well as the ground-based (GBT) samples, were shorter than intended.

Post-flight microstructural evaluation and microhardness studies were performed on the SPAR 2 and GBT samples. When the results were coupled with those from the SPAR 1 experiment, the following conclusions were reported:

(1) Low-gravity processing resulted in samples with no internal pores. Earth-processing resulted in ingots containing internal pores.

(2) The gettering-dispersion mechanism was successful in producing cast dispersion-strengthened metal matrix composites which cannot be obtained on Earth. Segregation, resulting from density differences between the dispersoids and matrix, can be reduced or

avoided by low-gravity processing. The "% Area with Sufficient Dispersion" was (a) approximately 90% for the low-g samples and (b) approximately 50% for the 1-g processed material. The average size of the dispersoids was the same in both the SPAR 2 and 1-g samples (0.5 to 0.6 microns) which should be adequate to attain dispersion strengthening at elevated temperatures.

(3) The average hardness of the low-g samples was significantly more uniform than that of the Earth-processed material. The hardness values of the rocket samples were twice that of pure Mg and about 30% greater than (a) the 1-g samples and (b) commercial Mg-Th-Zr (HK31A) alloys subjected to similar thermal conditions.

(4) "The short-duration of low-g achievable in SPAR flights can be utilized to conduct furnace experiments at elevated temperatures, provided time-dependent mechanisms and rates of heating and cooling do not complicate the experimental observations....

(5) "Sample preparation techniques are of paramount importance, especially when apparatus constraints and the characteristics of interfacing between sample, container, and the furnace may present problems not normally encountered in an Earth laboratory....

(6) "Known principles or theories pertaining to physical and chemical phenomena, rate processes, or phase equilibria can only be used as a rough guide at this embryonic stage of space processing technology. [See Reference (1) for a discussion of these theories.]

(7) "Past model space experiments involving nonreacting substances and oversimplified process parameters do not necessarily offer results or observations applicable to metallurgical experiments requiring high thermal energies. This experiment with Mg and ThO_2, in principle, approaches the meeting of requirements for a model experiment for future metallurgical experiments of dispersion-strengthening [of titanium] at low-g." (1, p. VII-48)

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Powder Metallurgy, Model Materials, Melt and Solidification, Casting, Metallic Matrix, Reaction Kinetics, Gettering Dispersion Mechanism, Dispersion Strengthening, Particle Formation, Stability of Dispersions, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Precipitation, Interface Physics, Inclusion and/or Rejection of Particles, Pores, Porosity, Oxidation, Density Difference, Thermal Soak, Quench Process, Solidification Rate, Sample Microstructure, Hardness, Material Strength, Incomplete Sample Processing

Number of Samples: two
Sample Materials: Mg-9.2 wt.% Th-3.2 wt.% MgO (2 vol.% ThO_2 after gettering; see the experiment summary above)
(Mg*Th*O*)
Container Materials: "POCO" graphite

Experiment/Material Applications:
"The ability of the Th to getter oxygen from MgO and to precipitate out as [a uniform distribution of] heavy inert ThO_2 particles in a light Mg matrix [while in the liquid state] is considered important in future space processing of dispersion-strengthened, high strength-to-weight ratio metals or alloys which contain difficult-to-remove oxygen impurity." (1, p. VII-3)

These metal matrix composites cannot be manufactured on Earth because of the significant difference in density and the reactive nature of the matrix metal.

See also Raymond, SPAR 1.

The specific reasons why argon was placed in the sample containers are detailed in the above experiment summary.

References/Applicable Publications:
(1) Raymond, L. and Ang, C. Y.: Casting Dispersion-Strengthened Composites at Zero Gravity. In Space Processing Applications Rocket Project, SPAR II- Final Report, NASA TM-78125, pp. VII-1 - VII-51, November 1977. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Naumann, R. J. (editor): Casting Dispersion-Strengthened Composites at Zero Gravity. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, NASA TM-78217, January 1979, pp. 7-8. (post-flight)

(4) Input received from Principal Investigator L. Raymond, August 1993.

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Experiment Origin: USA
Mission: SPAR 1
Launch Date/Expt Date: December 1975
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Directional Solidification Furnace
Builder of Processing Facility: Grumman Aerospace Corporation, Bethpage, New York

Experiment:
Uniform Dispersions of Crystallization Processing (74-15)

This SPAR 1 sounding rocket experiment was the first in a series of investigations designed by Uhlmann et al. to study the low-gravity interaction of dispersed particles with an advancing solidification front. The experiment "...was not intended to provide a critical or definitive evaluation of the physical phenomena. Rather, it represented a piggyback experiment which was intended primarily to provide insight into the characteristics of the research rocket flights and experimental variables which will be critical in future, definitive flights. Beyond this, it was intended to demonstrate within the time limitations of research rocket flights some important features of the interaction between second phase particles and a solidification front." (1, p. III-3)

Prior to the SPAR 1 mission, a naphthalene sample containing a combination of zinc particles and two types of glass beads was prepared. It was reported that (1) the Zn particles were 2 to 7 microns in diameter, and (2) the glass beads were (a) soda-lime-silicate glass (2 to 80 microns in diameter) and (b) lead-alkali-silicate glass (approximately 80 microns in diameter). The concentration of each particle type was about 1/3000 by volume.

The furnace hardware consisted of an aluminum block with cartridge heaters at both ends. A glass tube, containing the sample material, was placed in the aluminum block prior to the rocket launch. "The cartridge heaters were used to establish the temperatures of the ends of the aluminum block at 115 ^{o}C and 65 ^{o}C, respectively, with a temperature gradient of approximately 7.8 ^{o}C/cm between two ends. At the beginning of the experiments (upon launch in the case of the rocket experiments), the heaters

were turned off and the aluminum block was allowed to cool at a rate determined by convection in the ambient air and conduction through a thermal path from the low temperature end of the block to a heat sink." (1, p. III-13) A viewing port through the Al block and a light source above the sample allowed 35 mm photographic recording of the solidification process (1 frame per second, magnification of 1/2 X).

Post-flight examination of the flight photographs revealed "...the appearance of a vortex-like spiral in the molten naphthalene above the crystal-liquid interface. The spiral is apparently delineated by particles in the melt and very likely reflects the effects of fluid motion during the launch and despin phases of the flight. Any such vortex which existed during the despin phase apparently was damped out rather rapidly in the low-gravity period of the flight, leaving only an array of particles to mark its existence." (1, p. III-21)

Further analysis of the photographs indicated that the solidification rate of the material experienced an abrupt change. For the first 60 seconds of data recording (initiated 75 seconds into the flight) the solidification rate was about 2 microns per second. This was followed by a solidification rate of approximately 6 microns per second. The reasons for this change had not been established at the time available references were published but it appeared likely that it was due to thermal lag in the system.

Microstructural analysis of the flight sample revealed that rejection of the particles by the solidification interface had occurred. (Reference (1) provides a discussion of this phenomenon.) A large pile-up of particles was pushed by the interface over lengths of millimeters. Discrete clumps of particles, their location in the sample, and the curved shape of the interface at the beginning of data acquisition "...very likely reflect the initial distribution of particles prior to launch and the complex fluid motions which took place after launch. Despite the extensive and complex motion in the period after launch, good evidence has been provided for the absence of significant motion in the liquid during the microgravity period of the experiment." (1, p. III-36)

These results have led to several significant insights into various factors (material and environmental) which are essential in understanding the phenomenon from rocket flights:

(1) "A meaningful determination of the effects of important experimental variables on the behavior of second-phase particles at the solidification front requires careful and detailed control of experimental conditions as well as a carefully selected range of

solidification conditions....

(2) "The sample chamber should be relatively thin (but thick relative to the diameter of the largest particles being studied), and should be constructed with flat walls. This will permit better control of the solidification conditions and better determination of the interface shape. It would also permit effective photographic recording at reasonable magnifications.

(3) "Provision should be made for photographic recording of the solidification process at sufficiently high magnifications that individual particles can be seen interacting with the interface. This will provide a much better understanding of the interactions, and permit a better determination of the critical velocity for trapping.

(4) "The sample should not be melted prior to launch, but melting should be carried out in the microgravity environment. This will avoid the problems associated with highly nonuniform distributions (clumps) of particles.

(5) "The total concentration of particles should be kept small (0.1 percent or less), and a single size and type of particle should be investigated in a given run. This will avoid complications associated with pile-ups and with large particles pushed by small particles.

(6) "The sample materials should be partially degassed prior to insertion in the sample chamber. A small concentration of air bubbles generated at the interface should provide valuable information for the post-flight analysis, but should avoid problems associated with copious generation of gas at the interface.

(7) "For sample capsules in which photographic recording at magnification is not provided, careful recording should be taken of the thermal conditions, and the thermal conditions must be carefully controlled. This will permit meaningful results to be obtained from post-flight analysis of the solidified samples." (1, p. III-38)

Key Words: Composites with Solid Particles, Organic Systems, Transparent Liquids, Model Materials, Melt and Solidification, Directional Solidification, Thermal Gradient, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Bubbles, Bubble Formation, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Convection, Container Effects, Container Shape, Solidification Rate, Interface Shapes, Sample Microstructure, Rocket Motion, Acceleration Effects

Number of Samples: one
Sample Materials: Naphthalene sample containing (1) soda-lime-silicate glass beads, (2) lead-alkali-silicate glass beads, and (3) zinc particles
(Pb*Si*O*Zn*)
Container Materials: Glass

Experiment/Material Applications:
Naphthalene was selected for this experiment because it had the most desirable combination of properties for a sounding rocket experiment: (1) at the slow growth rates required by the experimental apparatus, it grows a smooth, faceted interface which does not break down to dendritic or needle-like morphologies, (2) the critical velocity (velocity at which particles are incorporated by the solidification interface) exceeds 10 microns/second (expected rate for flight furnace), and (3) other scientists have studied this material.

The zinc and glass particles were selected due to results from ground-based, critical velocity tests (see Reference (1)).

References/Applicable Publications:
(1) Uhlman [sic], D. R.: Uniform Dispersions of Crystallization Processing. In Space Processing Applications Rocket Project Spar I Final Report, NASA TM X-3458, pp. III-1 - III-40, December 1976. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

(3) Uniform Dispersions by Crystallization Processing. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 9-10. (post-flight)

(4) Uhlmann, D. R., Aubourg, P. A., and Joiner, B.: Multiphase Dispersions by Crystallization Processing. XXI COSPAR Plenary Meeting, Innsbruck, Austria, May 29-June 10, 1978.

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Experiment Origin: USA
Mission: SPAR 4
Launch Date/Expt Date: June 1977
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Apparatus which consisted of (1) seven cell assemblies, (2) a temperature controller for the top and bottom of the cells, (3) control electronics, and (4) a camera for photographing one of the cells.
Builder of Processing Facility: Unknown, possibly MIT Draper Labs, Cambridge, Massachusetts

Experiment:
Uniform Dispersions of Crystallization Processing (74-15)

When a solidification front encounters a second-phase particle, the particle will either be pushed ahead of the front or engulfed by the front. The type of behavior observed is dictated by a number of factors: (1) velocity of the interface, (2) size of the particle, (3) shape of the particle, and (4) characteristics of the particle/solidification front interactions.

This SPAR 4 sounding rocket experiment was the second in a series of investigations designed by Uhlmann et al. to study the low-gravity interaction of dispersed particles with an advancing solidification front (see Uhlmann, SPAR 1). The specific objective of the experiment was to determine the critical velocity (the maximum velocity at which second-phase particles are pushed by an advancing solidification front) as a function of various parameters.

Prior to the SPAR 4 mission, seven samples of d-camphor containing either (1) glass particles or (2) a mixture of glass and nickel particles were prepared. The particle sizes for these low-gravity experiments were selected such that, during ground-based experiments the smaller particles were easily pushed by the solidification front while the larger particles were not pushed by the front (see Reference (1) for particle size range). Each sample was contained in its own fused silica cell.

"The flight apparatus consist[ed] of seven sample cell assemblies, a temperature controller for both top and bottom of each of the seven cells, the necessary control electronics, and a camera and lens assembly to photograph one of the seven cells [sample cell no. 7]. A Nikon camera with a 250 exposure back... [was]... used to photograph... [cell no. 7]... at approximately 17X using a 19 mm Nikkor lens at approximately 50... [frames/minute]...." (1, p. II-15)

Four minutes prior to launch, the top heater block of each cell was preheated to 176 ^{o}C and the bottom heater block of each cell was preheated to 173 ^{o}C. Seventy-four seconds after launch, the top heater block temperature was increased to 197 ^{o}C and the bottom heater block temperature was decreased to 164 ^{o}C. This caused a melt-back of the samples and established a stable solidification interface with a gradient of approximately 60 ^{o}C/cm. Slow, controlled solidification was initiated at 119 seconds after launch by adjusting the setpoints of the heater blocks. At 310 seconds after launch, the heaters were switched off to allow rapid sample freezing prior to re-entry.

Post-flight, the following information was obtained from the photographs of sample cell no. 7:

"(1) The samples were not melted before launch. Hence the samples retain their initial particle dispersions.

"(2) [During the mission,] The sample melted back to approximately the correct location, leaving the particles dispersed in the liquid.

"(3) Unfortunately, when the camphor was melted, the camphor evaporated from the sample cell through a small crack in the side of the cell.

<Note: This only occurred in cell no. 7. Post-flight inspection of all other cells indicated that they "survived without problems.">

"(4) The dispersed particles in the liquid disappear[ed] from the field of view of the photographs...." (1, p. II-15)

(The disappearance of the particles from the field of view (50 micron depth of field) was attributed to Marangoni convection which swept the particles away.)

Post-flight examination of the remaining six good samples indicated that the second-phase particles "...were no longer dispersed through the upper halves of the sample cells (which had been melted). Rather, the particles had fallen into pile-ups at

the boundaries between the virgin samples and the portions which had been melted." (1, p. II-40) This distribution was compared to the expected Stokes' law terminal velocity for the particles. It was determined that "...residual microgravity levels could not have caused the distribution of particles observed in the recovered samples." (1, p. II-42)

Comparison of the particle distribution in the cells to the orientation of the rocket as it came to rest on the ground (after re-entry) was performed. It was concluded that "...the actual distribution of particles in the recovered samples... matches the distribution of particles expected if the [experimental] apparatus was recycled while resting on the ground after impact." (1, p. II-43) However, lack of post-flight telemetry data prohibited a definite conclusion.

Key Words: Composites with Solid Particles, Organic Systems, Transparent Liquids, Model Materials, Melt and Solidification, Directional Solidification, Thermal Gradient, Particle Dispersion, Particle Distribution, Particle Motion, Particle Velocity, Particle Size Distribution, Solid/Liquid Dispersion, Solid/Liquid Interface, Interface Stability, Sedimentation, Interface Physics, Inclusion and/or Rejection of Particles, Stokes Flow, Marangoni Convection, Acceleration Effects, Solidification Rate, Solidification Front Physics, Sample Evaporation, Gas Leakage, Processing Difficulties

Number of Samples: seven
Sample Materials: All seven samples had a d-camphor ($C_{10}H_{16}O$) organic matrix. Six of the samples contained glass beads and one sample contained a mixture of glass beads and nickel particles.
(C*H*O*, C*H*O*Ni*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
The entropy of fusion can have a significant effect on the solidification interface morphology and characteristics of the solidification process. Therefore, it was necessary to use a low entropy of fusion material such as d-camphor. This material also exhibits particle pushing behavior at reasonable solidification rates.

References/Applicable Publications:

(1) Uhlmann, D. R.: Experiment 74/15 Flown on SPAR 4. In Space Processing Applications Rocket Project SPAR IV- Engineering Report (Final), NASA TM-78235, pp. II-i - II-46, January 1980. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

(3) Uniform Dispersions by Crystallization Processing. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 9-10. (post-flight)

(4) Uhlmann, D. R., Aubourg, P. A., and Joiner, B.: Multiphase Dispersions by Crystallization Processing. XX1 COSPAR Plenary Meeting, Innsbruck, Austria, May 29-June 10, 1978.

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Experiment Origin: USA
Mission: SPAR 5
Launch Date/Expt Date: September 1978
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Apparatus which consisted of (1) seven cell assemblies, (2) a temperature controller for the top and bottom of the cells, (3) control electronics, and (4) a camera for photographing one of the cells.
Builder of Processing Facility: Unknown, possibly MIT Draper Labs, Cambridge, Massachusetts

Experiment:
Uniform Dispersion by Crystallization (74/15)

This SPAR 5 sounding rocket experiment was the third in a series of investigations designed by Uhlmann et al. to study the low-gravity interaction of dispersed particles with an advancing solidification front (see Uhlmann, SPAR 1, SPAR 4). The specific objective of the experiment was the same as the earlier SPAR 5 experiment: to determine the critical velocity (the maximum velocity at which second-phase particles are pushed by an advancing solidification front) as a function of various parameters.

Prior to the SPAR 5 mission, seven samples of d-camphor containing either (1) glass particles (five samples) or (2) nickel particles (two samples) were prepared. The particle sizes for the low-gravity experiments were selected such that, during ground-based experiments, the smaller particles were easily pushed by the solidification front but the larger particles were not pushed by the front (see Reference (1) for particle size range). Each sample was contained in its own fused silica cell.

It appeared that the experimental apparatus was the same as that used during the earlier SPAR 4 flight experiment (see Reference (1) for hardware details). Cell no. 7 was equipped with a camera to allow photographic recording of the solidification process (17.7X magnification, about 50 frames/minute).

Prior to rocket launch, "Two cell failures were observed during the filling of the cells for the SPAR 5 flight (one of which was intended for position 7) [see Reference (1) for cell filling

procedure]. In both cases, the failure took the form of the neck snapping off as a result of mechanical misalignment. Two spare cells were available to replace the two broken cells. Unfortunately, one of the spare cells apparently had a very small crack, as a very small leak was observed after the cell was filled with camphor. Since no additional replacement cell was available, the cell with the very small crack was flown (in position 1)." (1, p. II-20) <Note: Minor difficulties associated with calibration and telemetry are also discussed in Reference (1).>

Four minutes prior to launch, the top and bottom heater blocks of each cell were the cells were preheated to 170 ^{o}C. Eighty-nine seconds after launch (low-gravity conditions achieved), the top heater block temperature was increased to 193 ^{o}C and the bottom heater block temperature was decreased to 163 ^{o}C. This resulted in a melt back of the samples and established a stable solidification interface with a gradient of about 55 ^{o}C/cm. Slow, controlled directional solidification was initiated at 140 seconds after launch by adjusting the set-points of the heater blocks. At 300 seconds after launch, the heaters were switched off to allow sample freezing prior to re-entry.

Post-flight examination of the photographs from cell no. 7 led to the following conclusions:

(1) The sample in cell no. 7 did not melt prior to launch; therefore, the initial particle dispersion was retained prior to melting and resolidification under low-gravity conditions. It was inferred that this condition was also true for all other samples.

(2) The camphor was melted under low-gravity conditions, leaving the particles dispersed throughout the liquid.

(3) The interface melted back to a position within the field of view of the camera. However, the melt-back was not as far as desired (programmed).

Examination of the flight-processed samples (see Reference (1) for detailed discussion of each sample) indicated that "...particle pushing by a crystal-liquid interface does occur under microgravity conditions. The results also indicate that particle pushing in microgravity occurs with similar characteristics to those found by solidifying the same combinations of materials in 1-g." (1, p. II-48)

Key Words: Composites with Solid Particles, Organic Systems, Transparent Liquids, Model Materials, Melt and Solidification, Directional Solidification, Thermal Gradient, Particle Dispersion, Particle Distribution, Particle Motion, Particle Velocity, Particle Size Distribution, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Interface Stability, Interface Physics, Inclusion and/or Rejection of Particles, Stokes Flow, Solidification Rate, Solidification Front Physics, Processing Difficulties

Number of Samples: seven
Sample Materials: All seven samples had a d-camphor ($C_{10}H_{16}O$) organic matrix. Five of the samples contained glass beads and two of the samples contained nickel particles.
(C*H*O*Ni*, C*H*O*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
See Uhlmann, SPAR 4.

References/Applicable Publications:
(1) Uhlmann, D. R.: Technical Report on Experiment 74/15. In Space Processing Applications Rocket Project SPAR V Final Report, NASA TM-78275, pp. II-i - II-49, August 1980. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt. Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish TEXUS Experiment Module: two ellipsoidal mirror furnaces
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Metal Alloy With Oxide Inclusions

This TEXUS 1 experiment was the first in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites. The specific emphasis of the investigation was to examine composites with solid particles. The experiment was part of a preliminary research program designed to prepare for future Spacelab experiments.

During the mission, two samples of an Al-33 wt.% Cu alloy (eutectic composition) were directionally solidified. Both samples had a 2 vol.% distribution of alumina particles (10-100 microns in size).

Reference (4) indicated that either one or both of the samples did not completely melt. However, it was reported that no repulsion of the alumina particles occurred. This result was attributed to the formation of an oxide network.

No further details of the objectives, experimental setup, or results could be located in the available publications.

Key Words: Composites with Solid Particles, Ternary Systems, Eutectics, Dispersion Alloys, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Inclusions, Dispersion Strengthening, Incomplete Sample Processing

Number of Samples: two
Sample Materials: Al-33 wt.% Cu containing alumina particles (Al*Cu*Al*O*)
Container Materials: unknown

Experiment/Material Applications:
The behavior of suspended particles (solids, liquids, gasses,) located in front of a moving solidification interface is important to several industrial practices. For example, during the zone melting process, it is important that impurities which are rejected from the melt (and become concentrated in the melt ahead of the solidification front) remain ahead of the front. Conversely, during the production of dispersion-hardened metals, it is important that the solidification front overtake the particles (rather than push the particles) ahead of the front.

If the behavior of such particles can be better understood and controlled then the following industrial advantages might be realized:

(1) the cleansing of metals of impurities during the solidification process

(2) the production of dispersion-hardened metals directly from the melt (which would have better mechanical properties over those produced through powder metallurgical techniques)

(3) the production of metallic foams from the melt (without changing the inital pore distribution).

This study was performed in a low-gravity environment to reduce the segregation between constituents of differing densities (melt and suspended particles).

The specific reasons why the Al-Cu eutectic alloy (with oxide dispersions) was selected for these experiments were not detailed in the available publications.

References/Applicable Publications:

(1) Neuschütz, D. and Pötschke, J.: Estarrung einter Legierung mit Oxideinschlüssen in TEXUS I. KRUPP Forshungsinstitut, Untersuchungsbericht Nr. UB 1017/78, April 1978, 2 pp.

(2) Neuschütz, D. and Pötschke, J: Untersuchung zur Tonerdevernetzung und Porösität in Proben aus AlCu33 + 1% Al_2O_3- Ergänzender Abschussbericht zum TEXUS I-Experiment "Estarrung einer Legierung mit Oxideinschlüssen", Krupp Forschungsinstitut, 1979.

(3)Pötschke, J. and Hohenstein, K.: Preparation of Dispersed Alloys under Microgravity Conditions. In Acta Astronautica, Vol. 9, No. 4, pp. 261-264, 1982. (post-flight)

(4) Input received from Principal Investigator J. Pötschke, September 1989.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 2
Launch Date/Expt. Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment, Swedish Module
Processing Facility: Swedish TEXUS Experiment Module: two ellipsoidal mirror furnaces (MF 5 and MF 6)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Metal Alloy with Oxide Inclusions

This TEXUS 2 experiment was the second in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1). The emphasis of the investigation was to examine composites with solid particles. The experiment was part of a preliminary research program designed to prepare for future Spacelab experiments.

The specific objectives of the TEXUS 2 experiment were to:

(1) Observe the behavior of oxide particles at the solidification front (i.e., the particles pushed by the solidification front or overgrown by the crystal).

(2) Determine if the structure of the eutectic is influenced by the particles.

(3) Determine if the absence of gravity influences the formation of the eutectic and/or the particle suspension.

Prior to the flight, two samples of an Al-33 wt.% Cu alloy (eutectic composition) containing 1 wt.% alumina particles were prepared for the study. (Seventy-five percent of the alumina particles were between 20 and 100 microns in diameter and the remaining 25% were less than 20 microns.) Each of the 7 mm diameter, 12 mm long specimens was contained within a boron nitride crucible. The samples were prepared by powder metallurgical techniques, extruded, and then annealed (see Reference (1) for complete sample preparation details).

During the low-gravity portion of the TEXUS flight, each sample was processed in an ellipsoidal mirror furnace (MF 5 and MF 6). Reference samples were processed on Earth for comparison.

Post-flight examination of the thermal data and rocket samples revealed that certain procedural anomalies had occurred. "The temperature of the furnace MF 5 was programmed for a longer solidification period [than actually occurred]. The specimen was liquid for 25 sec. and attained 570 ^{o}C for a short time. It solidified for 55 sec." (1, p. 166) Further, a portion of the sample processed in MF 6 "...leaked out of a crevice of the cracked crucible into the furnace interior and solidified there in drops.... Owing to the defective furnace regulation the rest of the specimen in the crucible was subjected to three temperature changes between 550 and 670 ^{o}C before it solidified in about 10 sec." (1, p. 166)

Post-flight metallographic examination of the two samples led to the following conclusions:

- The samples should be prepared using powder-metallurgical techniques with the removal of any trapped gasses. A better suspension of particles will be obtained if the particles have a low affinity for gasses (such as oxygen, nitrogen, or hydrogen). The netting of the alumina particles, which may occur during sample preparation, can be removed by extrusion-moulding.

"- During the test period of about 90 sec. the alumina suspension was stable, i.e. no segregation was observed. Before a similar Spacelab experiment[,] tests must be carried out over longer periods....

"- The displacement of the alumina particles by the eutectic cells is observed in some places but in the majority of cases there is no connection between structure and alumina arrangement. An objective evaluation is made extremely difficult by the irregular arrangement of the eutectic cells and the alumina superimposed on the micrograph. Quantitative results can only be attained by using a pure base metal which exhibits directional solidification with a microscopic plane phase boundary. <Note: At the time this paper was written, such an experiment was anticipated for Spacelab.>

"- The structure formation is not affected by the alumina particle suspension. From this it can be presumed that the alumina will not alter the solidification process of copper in the Spacelab experiment either, which means that the preliminary tests on solidification carried out under 1 g conditions are transferable." (1, p. 160)

<Note: T. Carlberg (see Reference (2)) evaluated these TEXUS 2 flight specimens and concluded that the alumina particles substantially affected the solidification behavior by acting as nucleation sites, resulting in a finer eutectic structure.>

"- Gravity has no perceivable effect on the eutectic structure. A quantitative evaluation of the number and size of the eutectic cells is, however, not possible because the alumina masks the structure to an inadmissibly large extent." (1, p. 160)

Key Words: Composites with Solid Particles, Ternary Systems, Eutectics, Dispersion Alloys, Powder Metallurgy, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Motion, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Inclusions, Nucleation, Sample Microstructure, Liquid Leakage, Drops, Thermal Environment More Extreme Than Predicted, Incomplete Sample Processing, Furnace Malfunction

Number of Samples: two
Sample Materials: Al-33 wt.% Cu, containing alumina (1 wt.%) particles (less than or equal to 100 micron dia.)
(Al*Cu*Al*O*)
Container Materials: boron nitride
(B*N*)

Experiment/Material Applications:
See Neuschütz, TEXUS 1.

References/Applicable Publications:
(1) Pötschke, J. and Neuschütz, D.: Solidification of an Alloy with Oxide Inclusions. In Shuttle/Spacelab Utilization Final Report, Project TEXUS II, 1978, pp. 158-177. (post-flight)

(2) Carlberg, T.: Solidification of Some Eutectic Alloys Under Microgravity Conditions. In Proceedings of the 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 233-243. (post-flight)

(3) Input received from Principal Investigator J. Pötschke, September 1989.

(4) Alloys with Oxide Inclusions. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 288-289. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3
Launch Date/Expt. Date: April 1980
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01
Builder of Processing Facility: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Metal Oxide Dispersion Alloy Suspension (Metal Alloy with Oxide Dispersions)

This TEXUS 3 experiment was the third in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2). The emphasis of the investigation was to examine composites with solid particles. The experiment was part of a preliminary research program designed to prepare for future Spacelab experiments.

The specific objectives of this TEXUS 3 study were to (1) determine the stability of a suspension of particles in molten copper (under low-gravity conditions) and (2) determine if the particles were displaced by growing crystals.

Prior to the flight, three samples were prepared. Two of the samples (4.6 mm diameter, 45 mm long) were comprised of copper with a suspension of alumina particles. The particles were spherical with a diameter of less than 100 microns. (Most particles had a diameter between 20 and 63 microns.) The third sample (5.4 mm diameter, 45 mm long) was comprised of copper with a suspension of Mo particles. The Mo particles were 30 microns in diameter. The copper/alumina samples were each contained in a sintered corundum crucible (0.5 mm wall thickness). The copper/Mo sample was coated with a 50 micron thick alumina skin to permit skin-technology processing. (See Sprenger, TEXUS 1 for a discussion of skin technology.)

The samples were melted and directionally solidified in furnace C of the TEXUS Experiment Module TEM 01-2. "The samples were arranged at an angle of 120 degrees parallel to each other and asymmetrically to the isothermal center of the furnace in order

to bring about as high a degree of directional solidification from the cold end as possible." (1, p. 263).

Reportedly, because of a rocket despin failure, TEXUS 3 did not achieve the desired low-gravity level. It was reported in Reference (1) that the gravity level was 0.2 g.

Post-flight examination of the samples revealed that the unexpectedly high g-level resulted in a non-uniform distribution of particles throughout all three samples. It was also noted that the skin supporting the copper molybdenum sample "...withstood the test period of about 10 min and failed only after solidification when cooling gas (helium) was injected." (1, p. 263) <Note: Typically, a TEXUS sounding rocket flight lasts about 7 minutes. It is not clear to the editor what is meant by "...the test period of 10 min....">

Further information describing this experiment could not be located at this time. <Note: Reference (2) (as listed below) could not be attained at this time.>

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Motion, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Coated Surfaces, Skin Technology, Skin Casting, Thin Films, Crystalline Structure, Quench Process, Rocket Motion, Acceleration Effects, Rocket Despin Failure

Number of Samples: three
Sample Materials: copper with alumina particles (two samples); copper with molybdenum particles (one sample)
(Cu*Al*O*, Cu*Mo*)
Container Materials: copper-alumina samples: sintered corundum (0.5 mm thick); copper-molybdenum sample: 50 micron thick alumina skin
(Al*O*)

Experiment/Material Applications:
See Neuschütz, TEXUS 1.

References/Applicable Publications:
(1) Pötschke, J. and Hohenstein, K.: Preparation of Dispersed Alloys Under Micro-gravity Conditions. In Acta Astronautica, Vol. 9, No. 4, pp. 261-264, 1982. (post-flight)

(2) Pötschke, J. and Hohenstein, K.: The Behaviour of an Alumina-Copper Suspension Under Microgravity Conditions (TEXUS III). Presented at the IAF XXXII Congr. Intern. Astronautical Federation, Rome, September 6-12, 1981, BMFT FB Final Report, January 1982, Report Number UB 003/82, BMFT Reference Number 01 QV 159.

(3) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

(4) Pötschke, J. and Rogge, V.: The Behavior of Suspended Particles at the Solidification Front of Copper. In Proceedings of the Norderney Symposium on the Scientific Results of the German Spacelab Mission D1, August 27-29, 1986, pp. 304-308. (post-flight; general discussion of TEXUS results)

(5) Input received from Principal Investigator J. Pötschke, September 1989.

(6) The Behaviour of Alumina-Copper Suspensions. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 290-291. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 3b
Launch Date/Expt. Date: April 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01
Builder of Processing Facility: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Metal Alloys with Oxide Inclusions

This TEXUS 3b experiment was the fourth in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3). The emphasis of the investigation was to examine composites with solid particles. The experiment was part of a preliminary research program designed to prepare for future Spacelab experiments.

As reported under Neuschütz, TEXUS 3, a rocket despin failure occurred during the first TEXUS 3 flight. As a result, the experimental payload was subjected to a gravity level of 0.2 g (instead of the desired gravity level of 10^{-4} g). Thus, this TEXUS 3b investigation was designed to repeat the TEXUS 3 experiment objectives. Those objectives were to (1) determine the stability of a suspension of particles in molten copper (under low-gravity conditions) and (2) determine if the particles were displaced by growing crystals.

Prior to the flight, three samples were prepared. Two were comprised of copper with a suspension of alumina particles. The third was comprised of copper with a suspension of Mo particles. The Cu/Mo sample was coated with a 50 micron thick alumina skin to permit skin technology processing. The experimental setup, sample selection, and processing procedures for the TEXUS 3b experiment were similar to those employed during the TEXUS 3 investigations. (See Neuschütz, TEXUS 3 for a more detailed discussion of these aspects of the experiment.)

Post-flight examination of the TEXUS 3b samples revealed that the suspensions of the alumina and molybdenum particles were stable in all three specimens. Metallographic examination of one of the

copper-alumina samples revealed "...the oblong cells from which the copper crystals [were] built up." (1, p. 264) <Note: Discussion of only one of the copper-alumina samples was provided in the literature available to the editors. (Reference (2), as listed below, was not available.)> Particles greater than 5 microns in diameter were uniformly dispersed throughout the copper matrix. However, "The pearl string shaped arrangement of the fine alumina particles (...[radius]...less than or equal to 5 microns) [was] clearly visible. They were displaced transversely the direction of main growth into the residual molten areas (30 ppm oxygen), as in this direction the rate of solidification [was] at its lowest." (1, p. 264) <Note: Reference (3) stated that these displaced particles were less than or equal to 10 microns.>

It was reported that because of the lack of well-defined solidification parameters (e.g., solidification rate, planar solidification front), quantitative evaluation of the samples was not possible.

It was also reported that "...the influence exerted by the remaining impurities and the different thermal conductivity of alumina and copper on the behavior of the particles at the interface must be clarified theoretically and experimentally." (1, p. 264)

<Note: A discussion of the copper/Mo sample was not presented in the publications available to the editors.>

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Motion, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Coated Surfaces, Skin Technology, Skin Casting, Thin Films, Quench Process, Solidification Rate, Planar Solidification Interface, Inclusions, Thermal Conductivity Measurements, Impurities

Number of Samples: three
Sample Materials: copper with alumina particles (two samples); copper with molybdenum particles (one sample)
(Cu*Al*O*, Cu*Mo*)

Crucible Materials: copper-alumina samples: sintered corundum (0.5 mm thick); copper-molybdenum sample: 50 micron thick alumina skin
(Al*O*)

Experiment/Material Applications:
See Neuschütz, TEXUS 1.

References/Applicable Publications:
(1) Pötschke, J. and Hohenstein, K.: Preparation of Dispersed Alloys Under Micro-gravity Conditions. In Acta Astronautica, Vol. 9, No. 4, pp. 261-264, 1982. (post-flight; discusses TEXUS 3 and TEXUS 3b)

(2) Pötschke, J. and Hohenstein, K.: The Behaviour of An Alumina-Copper Suspension Under Microgravity Conditions (TEXUS III). Presented at IAF XXXII Congr. Intern. Astronautical Federation, Rome, September 6-12, 1981, BMFT FB Final Report, January 1982, Report Number: UB 003/82, BMFT Reference Number: 01 QV 159. (post-flight; abstract only)

(3) Pötschke, J. and Rogge, V.: The Behavior of Suspended Particles at the Solidification Front of Copper. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 304-308. (post-flight; general discussion of TEXUS results)

(4) Input received from Principal Investigator J. Pötschke, September 1989.

(5) The Behaviour of Alumina-Copper Suspensions. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 290-291. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 5
Launch Date/Expt. Date: April 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal four-chamber furnace
Builder of Processing Facility: Unknown, possibly Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Metal Alloys with Oxide Inclusions

This TEXUS 5 experiment was the seventh in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b; Pötschke, TEXUS 4 (two experiments; Chapter 4)). The emphasis of the investigation was to examine composites with solid particles. The experiment was part of a preliminary research program designed to prepare for future Spacelab experiments.

The specific objectives of the TEXUS 5 study were to investigate (1) the behavior of particles at a plane solidification front (i.e., steep thermal gradient), (2) the behavior of rod-shaped alumina particles, and (3) the influence of a circular tantalum crucible on the solidifying system (as compared to the nearly circular alumina crucible used in previous experiments).

Prior to the mission, a copper-1 wt.% alpha-alumina sample was prepared. The copper contained less than 30 ppm of oxygen. "The alumina particles were smaller than 20 microns and mainly cylindrical with a length/diameter ratio of between 5 and 10." (1, p. 3) The sample (8 mm dia. by 50 mm long) was prepared by powder-metallurgical techniques and extruded, which resulted in the alumina, rod-shaped particles being aligned with the longitudinal axis of the sample. The sample was contained within a tantalum crucible with a wall thickness of 0.5 mm. Four tantalum thermocouples were "..evenly distributed on the outer circumference of the sample at a c-c distance [presumably, center-to-center distance] of 15 mm which determined the temperature of the sample." (1, p. 3)

During the rocket flight, the sample was directionally solidified in one of the chambers of the isothermal, four-chamber furnace. The furnace was contained in the TEXUS Experiment Module TEM 01-1. Directional solidification was initiated by the introduction of He gas at the base of the sample. The nominally observed time/temperature profile can be located in Reference (1). <Note: The discussion in Reference (1) indicates that only one sample was processed during the flight. However, in the figures from Reference (1), it appears that two samples may have been processed.>

Reportedly, the sample was only partially melted. Post-flight examination of the thermal data indicated that the sample experienced temperature gradients of 80 to 120 K/cm and a solidification rate of 0.05 cm/sec. It was reported that the solidification process was significantly different than that observed during the TEXUS 3 experiment in that a planar solidification front was formed during this study. Metallographic examination of the sample revealed that, in the melted portion, "...the rod-shaped alumina particles were arranged transversely to the direction of solidification, i.e. parallel to the solid/liquid phase boundary, [proving] beyond doubt that the alumina is repelled by the copper crystal into the melt....The assumption of Van der Waals interactive force, on which the theory of the behavior of particles at solidification fronts...is based, thus received strong support from the findings of this experiment." (1, p. 6)

Examination of the sample also revealed the presence of circular, evenly distributed gas channels which were not seen in earlier tests of this material. After examination of the tantalum crucible, it was concluded that these channels were due to the carbon within the tantalum which formed carbon monoxide. It was, therefore, concluded that tantalum crucibles are suitable only if they are completely carbon-free.

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Powder Metallurgy, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Motion, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Solidification Rate, Planar Solidification Interface, Rod Structure, Inclusions, Impurities, Crucible Effects, Material

Interaction with Containment Facility, Van der Waals Forces, Incomplete Sample Processing

Number of Samples: unknown, possibly one or two
Sample Materials: copper containing 1 wt.% alumina particles (the particles were less than 20 microns in diameter)
(Cu*Al*O*)
Container Materials: tantulum
(Ta*)

Experiment/Material Applications:
See Neuschütz, TEXUS 1.

References/Applicable Publications:
(1) Pötschke, J.: Behavior of Dispersions Under Reduced Gravity Conditions, Texus V. IAF, International Astronautical Congress, 33rd, Paris, France, September 27-October 2, 1982, 8 pp. (post-flight)

(2) Pötschke, J.: Stabilität einer Suspension von stäbchenförmingen Tonerdeteilchen in erstarrendem Kupfer unter Mikrogravitationsbedingengen (TEXUS V). Untersuchungsbericht UB-NR: 008/83, 1983, 1 p.

(3) Pötschke, J. and Rogge, V.: The Behavior of Suspended Particles at the Solidification Front of Copper. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 304-308. (post-flight, general discussion of TEXUS results)

(4) Input received from Principal Investigator J. Pötschke, September 1989.

(5) Copper-Alumina Suspensions. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 292-293. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Interaction Between an Advancing Solidification Front and Suspended Particles (1ES302)

This Spacelab 1 experiment was the tenth in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5; Pötschke, TEXUS 4 (two experiments; Chapter 4), TEXUS 6, TEXUS 7 (both Chapter 4)). The specific objectives of the experiment were to (1) study the incorporation of particles within the solidified composite and (2) investigate the resultant particle spacing and size distribution.

During the mission, a copper melt containing 1% aluminum oxide particles, was to be melted and resolidified under low-gravity conditions. Reportedly, no results could be obtained from the experiment because the sample did not melt.

No further information could be found which discussed (1) the experimental setup or (2) why the sample did not melt.

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Motion, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Incomplete Sample Processing

Number of Samples: one
Sample Materials: copper containing 1% alumina particles. (Cu*Al*O*)
Container Materials: unknown

Experiment/Material Applications:
See Neuschütz, TEXUS 1.

References/Applicable Publications:
(1) Input received from Principal Investigator J. Pötschke, September 1989.

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering Publication (acceleration measurements on Spacelab 1)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 9
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEM 01-1: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Metal-Metal Suspension

This TEXUS 9 experiment was the eleventh in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, Spacelab 1; Pötschke, TEXUS 4 (two experiments; Chapter 4), TEXUS 6, TEXUS 7 (both Chapter 4)). The emphasis of the investigation was to examine composites with solid particles.

Previous experiments (in this investigative series) concerning composites with solid particles were formulated to examine the behavior of alumina suspensions within a copper or copper alloy melt (except TEXUS 3 and TEXUS 3b which included copper-molybdenum samples). During this TEXUS 9 study, one or more copper-molybdenum samples were processed under low-gravity conditions in the Isothermal Heating Facility (IHF). The sample(s) consisted of copper metal containing 1 vol.% molybdenum particles (2-20 microns). Control sample(s) were also processed under 1-g conditions for comparison purposes.

Post-flight analysis of the sample(s) indicated that the molybdenum suspensions were stable. However, the sample(s) processed under 1-g conditions also exhibited an unexpected stable suspension. (The investigators expected the molybdenum particles to sink within the melt during the 1-g experiments.) The results from the 1-g processed sample(s) were attributed to a cross-linking of the particles (even though the Mo particles are wetted by the melt and the vol.% should be too low for this phenomenon to occur).

No further information describing the results of this experiment could be located at this time. <Note: Reference (1) was not available at the time this summary was written.>

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Motion, Particle Wetting, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles

Number of Samples: unknown
Sample Materials: copper containing 1 vol.% molybdenum particles (2-20 microns)
(Cu*Mo*)
Container Materials: unknown

Experiment/Material Applications:
See Neuschütz, TEXUS 1.

References/Applicable Publications:
(1) Pötschke, J.: The Behavior of Molybdenum Particles in the Solidification of Copper. BMFT Fb Final Report, April 30, 1985, Report Number UB 036/85, Reference Number 01 QV 018, 19 pp. (TEXUS 9, post-flight)

(2) Input received from Principal Investigator J. Pötschke, September 1989.

(3) Copper-Molybdenum Suspension. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 294-295. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal furnace. (One of four available chambers within the furnace was employed for this experiment.)
Builder of Processing Facility: ERNO, Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
Stability of Compound Mixtures/Powder Metallurgy (Composite Materials I: Liquid-Solid-Gas Systems)

<Note: Walter performed two experiments on TEXUS 1 which involved the TEM 01-1 experiment module. Details of the other experiment can be found in Chapter 17: "Systems Exhibiting a Miscibility Gap."> This TEXUS 1 experiment was the first in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-solid-gas systems during melting, thermal soak, and solidification. The specific objective of the experiment was to examine the mechanisms (and their relative importance) which drive component separation. These mechanisms not only include (1) sedimentation and buoyancy (gravity effects), but also (2) volume changes, (3) interparticle forces, (4) the motion of bubbles or droplets in a temperature gradient, (5) interaction of solid, liquid, and gaseous inclusions with an advancing solidification front, (6) wetting, (7) liquid spreading, and (8) coalescence. Low-gravity processing permitted closer examination of mechanisms often masked by overwhelming gravity effects.

Four composite-powder model systems, representing solid-liquid-gas mixtures, were chosen for the TEXUS 1 flight. These systems varied from one another in terms of density, fluid/fluid wetting angle, particle size, and pore volume properties. In all four systems, silver (Ag) was chosen as the matrix material and all components were statistically distributed.

Reportedly, sample I(1) consisted of "...a sandwich arrangement of [1 mm diameter] Al_2O_3 [alumina] spheres accurately positioned between silver discs with matching hemispherical impressions...." (2, p. 31). This "sandwich arrangement" was annealed (on Earth) in hydrogen at 800 ^{o}C and 1 atmosphere for 30 minutes. Sample I(2) consisted of Ag and Al_2O_3 spherical particles (100 to 200 microns in diameter). The sample had an Ag/Al_2O_3 volume ratio of 4:1, a 30% pore volume and was (1) slightly compacted and (2) annealed using the same treatment as Sample I(1). Sample I(3) was the same as Sample I(2) but with an Ag/Al_2O_3 volume ratio of 3:1 (30% pore volume). This sample was (1) slightly compacted and (2) annealed using the same treatment as Sample I(1). Sample I(4) consisted of W powder (250 to 315 microns in diameter) and Ag powder (100 to 200 microns in diameter). The sample had a W/Ag volume ratio of 3:1, a 30% pore volume, and was (1) slightly compacted and (2) annealed (on Earth) in hydrogen at 900 ^{o}C and 1 atmosphere for 30 minutes.

Each of the four samples was placed in a molybdenum crucible (TZM alloy). The crucibles were stacked in one stainless steel cartridge. <Note: The stainless steel cartridge also housed a fifth sample (a liquid-liquid-gas system sample (See Walter, TEXUS 1 (Chapter 17))).>

The samples were processed in one of the four chambers of the TEM-01 isothermal furnace. Reportedly, the cartridge was preheated to 850 ^{o}C prior to lift-off and held at this temperature during launch. Once microgravity conditions had been achieved ($<10^{-4}$ g), the samples were heated to 1100 ^{o}C. After 30 seconds at this temperature, the samples were cooled such that they were below 600 ^{o}C by rocket re-entry.

Analysis of similar samples remelted under 1-g conditions illustrated that a significant degree of separation occurred in each of the samples because of the large density differences between the compound elements. It was reported that there was also an unexpected amount of separation in the flight samples. A discussion of each of the flight samples was presented.

Sample I(1) was processed to enable detection of small changes in particle position caused by residual accelerations. There was a complete separation of components in this sample resulting in a silver core (without inclusions) surrounded by alumina particles located at the crucible wall.

Sample I(2) underwent an almost complete separation. Alumina particles, located at the periphery of the crucible, were surrounded by a large Ag core (formed by coalescence). Only a few islands of alumina particles were located in the Ag matrix.

Sample I(3) consisted of droplets of Ag formed by coalescence and surrounded by alumina spheres. Shrinkage was evident in samples I(2) and I(3). In sample I(4), there was much less separation. While a silver core surrounded by W particles resulted, a significant amount of W particles remained in the silver matrix. Shrinkage was also evident in this sample.

The TEXUS 1 re-solidified samples all had a low surface free energy configuration. This result was highly significant. It indicated that the driving forces for separation of such components in the liquid state were not restricted to buoyancy and sedimentation. It was also apparent that rearrangement of the components occurred very rapidly since melting, soak, and resolidification occurred within the 5-minute low-gravity period. The separation which occurred in sample I(1), I(2), and I(3) was due to (1) the poor wetting (theta = 140-160^{o}) between silver and alumina particles and (2) coalescence of Ag droplets. In the case of sample I(1) sedimentation and buoyancy caused by residual accelerations may have been a major factor for separation (an examination of Stoke's law, given the experimental parameters, shows that a particle drift of 1 to 2 mm was possible). However, in sample I(4), the Ag and W were only partially separated even though their density difference was larger than the density difference between alumina and Ag. This partial separation occurred because the wetting of W by liquid Ag (theta = 70-90^{o}) is much better than that of alumina. This result "...indicates that the key to the stability of solid-liquid-gas mixtures is the contact angle." (2, p. 35)

"These results indicated that separation can take place under microgravity conditions as well. The preparation of heterogeneous alloys or composites from powder mixtures is, therefore, not possible with mixtures having non-wetting components and pore volume or degassing materials." (4, p. 284)

Key Words: Composites with Solid Particles, Composites with Gases, Melt and Solidification, Powder Metallurgy, Multiphase Media, Model Materials, Ternary Systems, Separation of Components, Stokes Sedimentation, Stability of Dispersions, Density Difference, Drops, Drop Migration, Thermomigration, Sedimentation, Buoyancy Effects, Wetting, Particle Wetting, Contact Angle, Surface Energy, Liquid Spreading, Particle Coalescence, Drop Coalescence, Particle Size Distribution, Particle Motion, Inclusion and/or Rejection of Particles, Pores, Thermal Soak, Thermal Gradient, Solidification Front Physics, Liquid/Gas Interface, Solid/Liquid Interface, Solid/Liquid/Gas Dispersion, Solid/Liquid Dispersion, Volume Change, Sample Shrinkage, Acceleration Effects

Number of Samples: four
Sample Materials: See Experiment section above.
(Ag*Al*O*, W*Ag*)
Container Materials: Molybdenum crucible with outer stainless steel envelope
(Mo*)

Experiment/Material Applications:
Earlier low-gravity experiments indicated that sedimentation and buoyancy were not the only forces separating components of composite materials (see, for example (in this chapter), Kawada, Skylab SL-3 and SL-4; Uhlman, SPAR missions). Other mechanisms (as detailed in the above experiment summary) could contribute to separation. It was necessary, therefore, to determine the extent and relevance of each of these forces.

The materials used in this study were chosen for many reasons: (1) the melt temperatures of each were below that of the available maximum furnace temperature, (2) large density differences between materials could illustrate separation due to residual acceleration components, and (3) wetting and non-wetting powder combinations would indicate separation due to this material characteristic. In addition, (1) no chemical reaction or solubility of components would occur, (2) plasticity of matrix material (Ag) allowed density control, (3) control of surface contamination and oxidation was possible, (4) the vapor pressure at maximum temperature would be low, and (5) the powders were available in the required form (particle shape, etc.).

References/Applicable Publications:
(1) Walter, H. U.: Stability of Multicomponent Mixtures Under Microgravity Conditions. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, p. 245. (post-flight)

(2) Walter, H. U. and Ziegler, G.: Stability of Multicomponent Mixtures. In Shuttle/Spacelab Utilization Final Report, Project TEXUS, 1978, Technological Experiments in Micro-gravity, pp. 27-47. (TEXUS 1 and 2; this paper is the same as the Grenoble paper above)

(3) Walter, H. U. and Ziegler, G.: Rearrangement and Separation Processes During Liquid Phase Sintering Under Microgravity Conditions. In Proceedings of the European Sounding-Rocket, Balloon and Related Research with Emphasis on Experiments at High Latitudes, Ajaccio, Corsica, April 24-29, 1978, ESA SP-135, pp. 345-352. (post-flight)

(4) Composite Materials I. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 284-285. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 2
Launch Date/Expt Date: November 1978
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: ERNO, Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
Stability of Compound Mixtures/Powder Metallurgy (Composite Materials II: Liquid-Solid Systems)

<Note: Walter performed two experiments on TEXUS 2 which involved the TEXUS TEM 01-1 experiment module. Details of the other experiment can be found in Chapter 17: "Systems Exhibiting a Miscibility Gap."> This TEXUS 2 experiment was the second in a series of investigations designed by Walter et al. to explore the low-gravity stability of multicomponent liquid-solid systems during melting, thermal soak, and solidification (see Walter, TEXUS 1). The specific objective of the experiment was to examine the mechanisms (and their relative importance) which drive component separation.

Two-component systems (representative of liquid-solid systems) were examined. Reportedly, because the chosen samples had no pores or free surfaces "...[(1)] Marangoni-flow generated at liquid-gas interfaces, [(2)] flow induced by volume expansion of gaseous inclusions, [(3)] melt bridge formation and resulting forces on particles, and especially [(4)] capillarity effects and coalescence...." (2, p. 36) should not be significant when resolving separation mechanisms. (All of these factors influenced the results from the TEXUS 1 experiment (see Walter, TEXUS 1).)

Three powder samples (prepared to the theoretical density) were selected for study. Sample II(1) consisted of Ag (particle diameter of 250 to 315 microns) and W (particle diameter of 100 to 200 microns). The sample had an Ag/W volume ratio of 4:1. One wt.% Ni and 2 wt.% Cu were added to reduce the contact angle, theta (between Ag and W) to less than 5^{o}. Sample II(2) was the same as Sample II(1) without the Ni and Cu additions. Theta ranged between 70^{o} and 90^{o}. Sample II(3) consisted of Ag

(particle diameter of 100 to 200 microns) and Al_2O_3 in the form of sapphire (particle diameter of 0.5 and 0.2 mm). The sample had an Ag/Al_2O_3 volume ratio of 4:1 and theta ranged between 140^o and 160^o.

Pre-flight preparation of the TEXUS 2 samples involved certain de-gassing and compaction procedures which the TEXUS 1 samples did not undergo (see Reference (1) for details).

The sample materials were processed under low-gravity conditions in the TEM 01 isothermal furnace. The melt and solidification sequence was similar to the TEXUS 1 sequence. The procedure was slightly altered such that the cartridge was pre-heated to 600 ^oC prior to TEXUS 2 lift-off (it was heated to 850 ^oC for TEXUS 1).

Post-flight examination of the low-gravity processed samples led to the following results:

Sample II(1) had a completely homogeneous distribution of W particles throughout the Ag matrix. This homogeneity indicated that capillarity and coalescence were of no importance in this sample. Sedimentation caused by residual acceleration was absent because the time available during the rocket flight was too short for the 100-200 micron sized particles to move. In Sample II(2), "No wetting agents were added, and it was expected that despite the poor wetting conditions an identical sample as I(1) would be obtained [Sample I(#) refers to TEXUS 1 samples.] However, some of the silver melt was removed from the crucible during processing due to capillary action, it was found in the gap between crucible and outer stainless steel envelope. As a result, larger pores were formed in the sample, the resulting sample was identical with sample I(4)...." (2, p. 36)

Sample II(3) revealed that the elimination of voids is one of the most important requirements during the production of homogeneous composites. The smaller particles remained in the Ag matrix and only the larger particles accumulated on one side of the sample. This one-sided accumulation was attributed to buoyancy effects.

Key Words: Composites with Solid Particles, Melt and Solidification, Powder Metallurgy, Multiphase Media, Model Materials, Binary Systems, Separation of Components, Stability of Dispersions, Homogeneous Dispersion, Free Surface Elimination, Density Difference, Drops, Drop Migration, Particle Migration, Particle Motion, Inclusion and/or Rejection of Particles, Thermomigration, Sedimentation, Buoyancy Effects, Wetting, Contact Angle, Particle Coalescence, Pores, Capillary Forces, Capillary Flow, Thermal

Soak, Solidification Front Physics, Solid/Liquid Interface, Solid/Liquid Dispersion, Liquid Leakage, Acceleration Effects

Number of Samples: three
Sample Materials: See Experiment section above.
(Ag*W*Ni*Cu*, Ag*W*, Ag*Al*O*)
Container Materials: molybdenum crucible in stainless steel envelope
(Mo*)

Experiment/Material Applications:
Material systems very similar to those of Walter's TEXUS 1 experiment were chosen for this flight. However, in order to eliminate the effect of a gaseous phase on the stability of multicomponent systems, it was necessary to select a material (1) with a low vapor pressure (at the experimental temperatures) and (2) without an "inner surface". Thus, "...Al_2O_3 particles [used in TEXUS 1] prepared by sintering were replaced by sapphire spheres." (1, p. 246)

References/Applicable Publications:
(1) Walter, H. U.: Stability of Multicomponent Mixtures Under Microgravity Conditions. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 245-253. (post-flight)

(2) Walter, H. U. and Ziegler, G.: Stability of Multicomponent Mixtures. In Shuttle/Spacelab Utilization Final Report, Project Texus, 1978, Technological Experiments in Micro-gravity, pp. 27-47. (TEXUS 1 and 2) (This paper the same as the Grenoble paper above.)

(3) Walter, H. U. and Ziegler, G.: Rearrangement and Separation Processes During Liquid Phase Sintering Under Microgravity Conditions. In Proceedings of the European Sounding-Rocket, Balloon and Related Research, with Emphasis on Experiments at High Latitudes, Ajaccio, Corsica, April 24-29, 1978, ESA SP-135, pp. 345-352. (post-flight)

(4) Composite Materials II. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 286-287. (post-flight)

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Experiment Origin: Japan
Mission: TT-500A 8 (Materials Processing Flight #1)
Launch Date/Expt Date: September 1980
Launched From: Takesaki Launch Site on Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Two electric furnaces
Builder of Processing Facility: Ishikawajima Harima Industry Company, Japan

Experiment:
Ni-TiC Composite Alloy

Turbine engine components are subjected to high stress, high temperature environments. Components fabricated from ceramic-strengthened metal composites can withstand these severe environments. However, fabrication of such materials is difficult on Earth because gravity-induced convection and sedimentation affect composite strength properties.

This TT-500A 8 experiment was the first in a series of investigations designed by Takahashi to study the solidification of ceramic-strengthened metallic composites. The specific objective of the experiment was to prepare a composite material by melting, pressing, and solidifying under low-gravity conditions.

Prior to the mission, two samples were prepared. Sample 1 consisted of a nickel-molybdenum alloy (10 wt.% Mo) with titanium carbide whiskers (30 wt.%). The whiskers, which were 10-50 microns thick and 0.5-2.0 mm long, were plated with rhodium to prevent reaction with the Ni-Mo matrix at 1300 ^{o}C. The sample was prepared on Earth by (1) mixing the material with an aqueous solution of ammonium alginate (to make it "clayish" for ease of extrusion), (2) extruding the material through small cylindrical holes (to align the whiskers), (3) drying the material, and (4) heating the material at 1100 ^{o}C for 30 minutes (to evaporate the alginate).

Sample 2 consisted of a nickel-molybdenum alloy (10 wt.% Mo) with 1-2 micron diameter titanium carbide particles (25 wt.%). The sample was prepared by (1) mixing the Ni, Mo, and TiC powders, (2) pressing the blended powder into cylindrical form (500 MPa), and (3) heating at 1100 ^{o}C for 30 minutes under a vacuum of 10^{-5}

Torr.

Each sample was contained in a carbon crucible. The crucible contained a carbon piston used for pressing the sample material. The samples were pressed during processing to eliminate voids which would normally be created when the material was heated. Each of the crucibles was placed in an electric furnace for processing.

During the 6-minute rocket flight, the samples were (1) heated, (2) melted, (3) soaked, (4) pressed, and (5) cooled. Although a constant soak temperature of 1450 ^{o}C was intended, it was reported that "...sample [1] did not seem to be completely melted because the time elapsed to reach 1450 ^{o}C was behind... [schedule].... In the heating of [the] Ni-Mo-TiC particle sample [sample 2], the temperature rose to above 1900 ^{o}C.... So the sample rapidly expanded on account of the quick heating, then most of molten sample... flowed out of the crucible." (1, p. 222)

It was further reported that the desired gravity (10^{-4} g) level was not achieved because of a problem with the rocket depin system. "...the gravity levels were 8.0 x 10^{-2} g in the direction of pitch axis[,]... 6.0 x 10^{-2} g in yaw-axis, and... 7.5 x 10^{-4} g..." in the longitudinal axis. (1, p. 222)

Additional samples were similarly processed on Earth for comparison.

Post-flight examination of the samples included microphotography, microvicker's hardness tests, and X-ray microanalysis. The following results for the whisker and particle samples were reported:

Low-gravity whisker sample (Sample 1):
(a) It appeared that this sample was pressed and cooled after insufficient melting. Some voids were located in the sample.

(b) An increase in hardness values of the flight sample was indicated (the hardness value of the low-gravity sample was 101 before flight; 350 after low-gravity processing). Reportedly, the whisker sample had a lower hardness value than the low-gravity particle sample (Sample 2). This was "...considered to be due to the whisker reinforced sample preparation process being insufficient in temperature condition." (1, p. 223)

(c) The sample exhibited a vertical alignment of the TiC whiskers. An annular ring-like structure was present "...which show[ed] the growing process of whisker crystal by chemical vapour deposition...." (7, p. 749)

(d) X-ray microanalysis (EPMA, line scanning) illustrated "...that the Mo elements highly concentrate on the boundary of [the] Ni matrix and TiC whiskers...." (7, p. 749)

Low-gravity particle sample (Sample 2):

(a) The dispersion of TiC particles in this sample seemed to be uniform.

(b) As was observed in flight Sample 1, an increase in hardness between the before-flight sample and low-gravity sample was indicated (the hardness value was 150 before flight; 545 after low-gravity processing).

(c) X-ray analysis indicated that "...Mo was recognized to exist overlapping with the region of TiC particles...." (1, p. 223)

Analysis of the samples, considering such factors as sedimentation, random walk of the particles, and Marangoni forces led to the following conclusions:

(1) The migration of the particles (1-2 microns) in Sample 2, attributed to sedimentation, was considered negligible when a gravity level of 10^{-3} was taken into account. However, the larger size of the whiskers in Sample 1 resulted in "...a migration distance remarkably increased." (1, p. 224)

(2) Since the contact angle between molten Ni-Mo and TiC was regarded as zero or near zero, the tendency of the particles to coagulate by random walk was considered negligible.

(3) "...the molten specimen does not have [a] free surface since the surroundings of the specimen is limited by the wall of a crucible made of carbon. So it is expected that even if a gradient occurs in the surface tension, its movement is hindered by carbon wall and no flow occurs in the molten specimen." (1, p. 225)

Further details of the sample analysis are located in Reference (1). Quantitative and theoretical analysis of the various forces which affect the particle movement, such as floating (using Stokes' law), wetting between TiC and Ni, and random walk, were included in Reference (7).

<Note: References (4) and (5) were not available at the time this experiment summary was prepared.>

Key Words: Composites with Solid Particles, Dispersion Alloys, Ternary Systems, Reinforced Materials, Dispersion Strengthening, High Temperature Materials, Melt and Solidification, Metallic Matrix, Whiskers, Ceramics, Coagulation, Particle Dispersion, Particle Distribution, Particle Size Distribution, Wetting, Particle Wetting, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Sedimentation, Vapor Deposition, Buoyancy-Driven Convection, Stokes Flow, Marangoni Force, Interface Physics, Thermal Soak, Free Surface Elimination, Contact Angle, Voids, Hardness, Material Strength, Sample Microstructure, Piston System, Volume Expansion, Liquid Leakage, Thermal Environment More Extreme than Predicted, Rocket Motion, Acceleration Effects, Turbine Blade Applications, Incomplete Sample Processing

Number of Samples: two
Sample Materials: Sample 1: nickel-molybdenum alloy (10 wt.% Mo) with titanium carbide whiskers (30 wt.%); Sample 2: nickel-molybdenum alloy (10 wt.% Mo) with titanium carbide particles (25 wt.%)
(Ni*Mo*Ti*C*)
Container Materials: carbon ampoule (with carbon piston for applying pressure)
(C*)

Experiment/Material Applications:
Many types of whisker materials may be used for high-strength applications. However, it is difficult to produce these whiskers in an untangled state. TiC whiskers can be easily individualized by soaking in a solution.

Ni-Mo was used as the matrix material because (1) it wets well the TiC material well, (2) it has good heat resistance, and (3) it is easy to obtain and handle.

References/Applicable Publications:
(1) Takahashi, S.: Preparation of Ni-TiC Metal Composite System Under Low Gravity Conditions. In 2nd Joint Japan-Germany-ESA Symposium on Microgravity Research, Tokyo, March 25-26, 1985, Summary Report, pp. 219-225. (post-flight)

(2) Sawaoka, A. B. and Kanbayashi, A.: Recent Developments and Future Outlook in Japan. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 14-24. (post-flight)

(3) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. International Astronautical Federation, 34th International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary; post-flight)

(4) Takahashi, S., Ono, M., and Ueda, Y.: Distribution of Elements in Ni-Mo-TiC Alloy Processed Under Microgravity Conditions. Journal of Material Science, Vol. 25 (1990), pp. 2213-2220.

(5) Takahashi, S.: High Temperature Compression Strength of TiC Particle Dispersion Ni Alloy Processed on the Ground and Rocket. 1993 Powder Metallurgy World Congress (1993), Kyoto, Japan.

(6) Input received from Principal Investigator S. Takahashi, August 1993.

(7) Takahashi, S.: Preparation of Ni-TiC Metal Composite in Space. In 5th International Conference on Composite Materials ICCM-V, Sponsored by the TMS Composite Committee in San Diego, CA, July 29-August 1, 1985, edited by Harrigan, W. C., Jr., Strife, J., and Dhingra, A. K., pp. 747-754. (post-flight)

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Experiment Origin: Japan
Mission: TT-500A 9 (Materials Processing Flight #2)
Launch Date/Expt Date: January 1981
Launched From: Takesaki Launch Site on Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Three electric furnaces
Builder of Processing Facility: Ishikawajima Harima Industry Company, Japan

Experiment:
Ni-TiC Composite Alloy

This TT-500A 9 sounding rocket experiment was the second in a series of investigations designed by Takahashi to study the low-gravity solidification of ceramic-strengthened metallic composites (see Takahashi, TT-500A 8).

The specific experimental goals and equipment configuration of the experiment were not detailed in the available publications. However, the Principal Investigator briefly reported that (1) the objective of the experiment was "...to obtain void free and high density ratio samples which were uniformly dispersed by TiC particles" (Reference (2)) and (2) the TT-500A 9 processing facility was changed slightly after the TT-500A 8 mission. <Note: The changes to the facility were not detailed.>

Although the rocket was successfully launched, it was reported that "...the TT-500A rocket...[was]...not recovered because its floating system malfunction." (1, p. 2)

No further details could be located concerning (1) the floating system malfunction or (2) the evaluation of any real-time data.

<Note: Even though the rocket was not recovered, real-time data from the experiment may have been obtained during the flight. The Principal Investigator indicated that real-time data may have been kept "in NASDA.">

Key Words: Composites with Solid Particles, Dispersion Alloys, Ternary Systems, Reinforced Materials, Dispersion Strengthening, High Temperature Materials, Melt and Solidification, Metallic Matrix, Whiskers, Ceramics, Coagulation, Particle Dispersion, Particle Distribution, Particle Size Distribution, Particle Wetting, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Sedimentation, Buoyancy-Driven Convection, Interface Physics, Thermal Soak, Voids, Hardness, Material Strength, Turbine Blade Applications, Piston System, Payload Recovery System Failure

Number of Samples: three
Sample Materials: TiC particle dispersion strengthened Ni-Mo base alloys (specific compositions unspecified)
(Ni*Mo*Ti*C*)
Container Materials: carbon (graphite) ampoule (with carbon piston for applying pressure)
(C*)

Experiment/Material Applications:
See Takahashi, TT-500A 11.

References/Applicable Publications:
(1) Kajiwara, K., Matsuda, T., Shibato, T., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. International Astronautical Federation, 34th International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary; post-flight)

(2) Input received from Principal Investigator S. Takahashi, August 1993.

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Experiment Origin: Japan
Mission: TT-500A 11 (Materials Processing Flight #4)
Launch Date/Expt Date: August 1982
Launched From: Takesaki Launch Site on Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: One electric furnace
Builder of Processing Facility: Ishikawajima Harima Industry Company, Japan

Experiment:
Ni-TiC Composite Alloy

<Note: The following summary was written using Reference (2). This reference, however, did not specifically state that the objectives, experimental setup, and results described therein were from the TT-500A 11 mission. The Principal Investigator verified that this Reference is discussing results from both the TT-500A 8 flight (Sample 1, the whisker sample) and a single sample processed on the TT-500A 11 flight.>

This TT-500A experiment was the third in a series of investigations designed by Takahashi to study the solidification of ceramic-strengthened metallic composites (see Takahashi, TT-500A 8, TT-500A 9). The specific objective of the experiment was to melt, press, and solidify a composite material under low-gravity conditions.

Prior to the mission, one sample was prepared. The sample consisted of a nickel-molybdenum alloy (10 wt.% Mo) with 1-2 micron diameter titanium carbide particles (25 wt.%). <Note: Reference (2) indicated that 20 wt.% titanium carbide particles were employed. The Principal Investigator indicated that this value was actually 25 wt.%.> The sample was prepared on Earth by (1) mixing the Ni, Mo, and TiC powders, (2) pressing the blended powder into cylindrical form (200 MPa), and (3) heating at 1260 ^{o}C for 30 minutes under a vacuum.

The sample was contained in a carbon crucible. The crucible contained a carbon piston used for pressing the sample material. The sample was pressed during processing to eliminate voids which would normally be created when the material was heated. The crucible was placed in an electric furnace for processing.

During the rocket flight, the sample was (1) melted within 2 minutes after the start of heating, (2) soaked at 1450 ^{o}C for 2 minutes, and (3) cooled (the sample temperature was 700 ^{o}C 500 seconds after launch).

Post-flight metallographic analysis of the particle sample revealed that (1) the sample completely melted during the flight and (2) a uniform dispersion of TiC particles within the Ni-Mo matrix had occurred.

Quantitative and theoretical analysis of the various forces, such as floating (using Stokes' law), wetting between TiC and Ni, and random walk, which affect the particle movement were included in Reference (2).

Additional detailed information describing the sample analysis did not appear to be available at this time.

Key Words: Composites with Solid Particles, Dispersion Alloys, Ternary Systems, Reinforced Materials, Dispersion Strengthening, Powder Metallurgy, High Temperature Materials, Melt and Solidification, Metallic Matrix, Ceramics, Particle Dispersion, Particle Distribution, Particle Wetting, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Sedimentation, Buoyancy-Driven Convection, Stokes Flow, Surface Energy, Interface Physics, Thermal Soak, Voids, Material Strength, Sample Microstructure, Piston System, Turbine Blade Applications, Vacuum

Number of Samples: one
Sample Materials: nickel-molybdenum alloy (10 wt.% Mo) with titanium carbide particles (25 wt.%)
(Ni*Mo*Ti*C*)
Container Materials: carbon ampoule (with carbon piston for applying pressure)
(C*)

Experiment/Material Applications:
Ni-Mo was used as the matrix material because (1) it wets well the TiC material well, (2) it has good heat resistance, and (3) it is easy to obtain and handle.

References/Applicable Publications:

(1) Takahashi, S.: On the Behavior of Titanium Carbide Particles in Molten Nickel Alloy Under the Micro Gravity Conditions. In Proceedings of the 15th International Symposium on Space Technology and Science, Tokyo, 1986, pp. 543-548. (post-flight)

(2) Takahashi, S.: Preparation of Ni-TiC Metal Composite in Space. In 5th International Conference on Composite Materials ICCM-V, Sponsored by the TMS Composite Committee in San Diego, CA, July 29-August 1, 1985, edited by Harrigan, W. C., Jr., Strife, J., and Dhingra, A. K., pp. 747-754. (post-flight)

(3) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. International Astronautical Federation, 34th International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (post-flight)

(4) Input received from Principal Investigator S. Takahashi, August 1993.

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Co-Investigator(s): Yoel, D. (Payload Manager, Lead Engineer) (2), Moore, R. G. (Contributor/Customer) (3)
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Experiment Origin: USA
Mission: STS Launch #4, STS-004 (STS OFT-4, Columbia)
Launch Date/Expt. Date: June 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: College Student Experiment
NASA Get Away Special (GAS) Canister G-001
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-001: Utah State University, Logan, Utah/R. Gilbert Moore
Processing Facility: Small aluminum furnace
Builder of Processing Facility: A. M. Dalley (design and machining), Utah State University, Logan, Utah and other personnel at Utah State University, Logan, Utah

Experiment:
Composite Curing (Experiment Number 12-M)

This STS-004 experiment was one of ten investigations housed within the G-001 Get Away Special Canister during STS-004. Four other experiments (of the ten) were applicable to this this data base (see Alford, STS-004 (Chapter 18); Elwell, STS-004 (Chapter 12); Laher, STS-004 (Chapter 17); Thomas, T. L., STS-004 (Chapter 14)). The objective of the experiment was to demonstrate the curing of an epoxy resin-graphite composite.

Prior to the flight, a partially-cured composite sample was selected for the space investigation. During the mission, the sample was to be heated to 329 ^{o}F and held at this temperature for 70 minutes (allowing the resin to gel). Post-flight, the resultant composite strength and "...quality of wetting between the resin and graphite fibers" (1, p. 12) was to be compared to a similarly processed ground-based sample.

Reportedly, the experimental objectives could not be completed as planned. The heating apparatus, although operational, did not provide the necessary operating temperature. (The temperature of the GAS canister was lower than expected.) The Principal Investigator provided additional information on this anomaly: "A

shuttle/canister wiring malfunction delayed all experiment initiations. Meanwhile the shuttle was performing its 'cold soak' mission in which the bay was pointed into space and its temperature dropped significantly. When the experiments were finally activated, the starting temperature for the graphite composite furnace was so low that the batteries were depleted before achieving the necessary temperature for curing the composite." (12)

<Note: Chapter 17, "Systems Exhibiting a Miscibility Gap," contains another experiment involving the curing of epoxy resins (see Harris, "Elastomer-Modified Epoxy Resins Heater").>

Key Words: Composites with Solid Particles, Curing, Epoxy Resins, Fibers, Fiber Dispersions, Particle Dispersion, Suspension of Particles, Interface Physics, Wetting, Particle Wetting, Tensile Strength, Coated Surfaces, Material Strength, Thermal Environment More Extreme Than Predicted, Furnace Malfunction, Incomplete Sample Processing

Number of Samples one
Sample Materials: B-staged (partially cured) E-7k7/PW-70 epoxy-resin-graphite (manufactured by U. S. Polymetric, California)
Container Materials: aluminum furnace with Teflon™-coated release paper

Experiment/Material Applications:
Fiber-resin composites have applications in several material areas including structural components.

This experiment sought to investigate the effects of reduced gravity curing on (1) sample tensile strength and (2) wetting between graphite fibers and resin.

References/Applicable Publications:
(1) Yoel, D., Walker, S., Elwell, J., and Moore, G.: The First Getaway Special- How it was Done. Spaceworld, May 1983, pp. 9-16. (post-flight)

(2) STS-4 Fourth Space Shuttle Mission, NASA Press Kit, June 1982, p. 61. (preflight)

(3) Yoel, D. W.: Payload Integration of a Get Away Special Canister. American Institute of Aeronautics and Astronautics, Annual Meeting and Technical Display on Frontiers of Achievement, Long Beach, California, May 12-14, 1981, 5 pp. (preflight)

(4) The STS-4 Getaway Special. NASA Report PB82-10223, May 20, 1982. (preflight)

(5) Cargo Systems Manual: GAS STS-4, May 20, 1982, JSC-17645, pp. 4-1 - 4-4. (preflight)

(6) Overbye, D.: The Getaway Kids Shuttle Into History. Discover, September 1982. (post-flight)

(7) Yoel, D. W.: Analysis of the First Getaway Special Space Shuttle Payload. Thesis for M.S. in Physics, Utah State University, Logan, Utah, 1984.

(8) Moore, R. G.: Educational Implications of Getaway Special Payload Number One, IAF-81-293, XXXIInd International Astronautical Federation Congress, Rome, September 6, 1981. (preflight)

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

(10) "Get Away Special," NASA News, NASA MSFC, June 7, 1982.

(11) Transcripts of Press Conference at NASA MSFC with G-001 Student Experimenters and Sponsors, NASA, May 20, 1982.

(12) Input received from Principal Investigator A. Dalley, July 1993.

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Co-Investigator(s): Mishima, Y. (3)
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Experiment Origin: Japan
Mission: TT-500A 13 (Materials Processing Flight #6)
Launch Date/Expt. Date: August 1983
Launched From: Takesaki Launch Site on Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Electric furnace
Builder of Processing Facility: NASDA, Tokyo, Japan

Experiment:
Fabrication of C-Fiber/Al Composite

Future space structural building materials may include a metallic composite which exhibits a random three-dimensional array of high modulus, short carbon fibers bonded at contact points by an aluminum alloy coated on the fibers. The composite is highly porous (and of extremely low density) and would be an ideal material for space components.

A reduced gravity environment is necessary for the fabrication of such a composite because (in low-g), (1) the short fibers will remain in a random three-dimensional configuration (instead of collapsing to a gravity-driven, nearly two-dimensional configuration) and (2) the molten metal will not slip and separate in the direction of gravity because of density differences of the fiber and metal.

The specific objective of this TT-500A sounding rocket experiment was to fabricate an extremely low density, composite material exhibiting high stiffness characteristics.

Prior to the rocket flight, short carbon fibers of the order of 0.5 mm in length (coated with an Al-1 at% Tl alloy to a thickness of approximately 1 micrometer) were packed in a fused silica tube. The silica tube was configured in a graphite container and then placed in an electric furnace. During the rocket flight, the sample was heated above the melting temperature of the alloy.

Reportedly, "The maximum temperature was scheduled to be 1023 K for 2 min while the melting point of the aluminum alloy is about 943 K. Unfortunately, temperature control of the electric furnace was unsuccessful and the temperature went up to above 1473 K....

"Examination of the retrieved specimen shows... damage on the surface of the constituent fibres and the whole specimen was found quite brittle. This is probably due to the chemical reaction between the fibre and the aluminum alloy coating to form Al_4C_3 and due to the partial evaporation of the aluminum alloys both attributed to the extreme overheating. SEM observations, however, revealed the structure of the specimen to be a random three-dimensional configuration of the short fibres and the contact-point bonding was mostly successful making the entire specimen rigid, thereby encouraging future experiment.... [Further,] the aluminum alloy coatings are severely deteriorated, from which any mechanical property evaluation was considered meaningless." (1, p. 2765)

<Note: A more detailed description of the processing facility and its performance during the flight can be found in Reference (5).>

Key Words: Composites with Solid Particles, Metallic Matrix, High Stiffness Composites, Porous Material, Melt and Solidification, Fibers, Coated Fibers, Fiber Dispersions, Particle Wetting, Solid/Liquid Dispersion, Solid/Liquid Interface, Density Difference, Separation of Components, Interface Physics, Porosity, Space Structures, Material Strength, Thermal Environment More Extreme Than Predicted, Evaporation, Furnace Malfunction

Number of Samples: one
Sample Materials: 7-8 μm diameter, short carbon fibers of the order of 0.5 mm in length. The fibers were coated with Al-1 at% Tl alloy to a thickness of approximately 1 μm.
Container Materials: 10-inch diameter fused silica tube (Si*O*)

Experiment/Material Applications:
The application being pursued is the on-orbit fabrication of structural components (such as a beam).

References/Applicable Publications:

(1) Mishima, Y., Hori, M., Suzuki, T., and Umekawa, S.: Fabrication of Carbon/Fibre[sic] Aluminum Alloy Composite Under Microgravity. Journal of Materials Science, Vol. 21 (1986), pp. 2763-2766. (post-flight)

(2) Mishima, Y., Suzuki, T., and Umekawa, S.: Carbon Fiber/Aluminum Composite with Ultra Low Density and High Stiffness. In 2nd Joint Japan-Germany-ESA Symposium on Microgravity Research, Tokyo, March 25-26, 1985, Summary Report, pp. 229-238. (post-flight)

(3) Kimura, Y., Mishima, Y., Umekawa, S., and Suzuki, T.: Compatibility Between Carbon Fibre[sic] and Binary Aluminum Alloys. Journal of Materials Science, Vol. 19 (1984), pp. 3107-3114. (ground based results; preflight)

(4) Input received from Experiment Investigator Y. Mishima, August 1989.

(5) Shu Usuba, Ken-ichiro Tani, et al.: Thermal Characteristics of an Electric Furnace Under Micro-Gravity. In Jpn. J. Appl. Phys., Vol. 23 (1984), No. 8, pp. 1163-1164. (post-flight)

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Experiment Origin: Japan
Mission: TT-500A 13 (Materials Processing Flight #6)
Launch Date/Expt Date: August 1983
Launched From: Takesaki Launch Site on Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Image Furnace
Builder of Processing Facility: Unknown

Experiment:
PbO-B_2O_3/Diamond Composite

Reference (1) indicated that this composite material experiment was to be performed during the TT-500A 13 sounding rocket mission.

No publications (published either prior to or after the TT-500A 13 flight) could be located which specifically discussed the experimental objectives, setup, or results.

Key Words: Composites With Solid Particles, Melt and Solidification

Number of Samples: unknown
Sample Materials: unknown, appears to have been a PbO-B_2O_3/Diamond Composite
(Pb*O*B*)
Container Materials: unknown

Experiment/Material Applications:
Unknown

References/Applicable Publications:

(1) Sawaoka, A.: Japanese Effort Towards Materials Processing in Space. In Manufacturing in Space: Proceedings of the Winter Annual Meeting, Boston, Massachusetts, November 13-18, 1983, pp. 33-42. (overall discussion of Japanese low-gravity materials processing)

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Experiment Origin: Great Britain
Mission: TEXUS 10
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish Space Corporation (SSC) TEXUS Experiment Module Furnaces. <Note: Although it was not specifically stated in Reference (1) the furnaces used for this investigation may have been the same as those employed during Caton's TEXUS 7 experiment (see Chapter 17).>
Builder of Processing Facility: Swedish Space Corporation/ACR, Solna, Sweden

Experiment:
Manufacture of a Particle Dispersion

A melt containing a fine dispersion of insoluble particles "... could be produced by a powder processing route (e.g. Al-alumina fibres) or by rapid solidification processing of systems which the intermetallic compounds exhibit sluggish dissolution kinetics in the melt (e.g. Al-Zr, Al-Nb). Normally heating of this rapidly solidified material above the melting point of the matrix would rapidly be followed by coarsening of the distribution of aluminide particles with agglomeration and coalescence. This would be largely the result of convection-driven collision events.

"Under micro-gravity conditions, on the other hand, it would be expected that coarsening of the particle distribution would be minimal and there would be potential for retention of an ultrafine distribution of insoluble particles in a bulk artefact[sic]- an attractive objective for dispersion-strengthened systems. However, apart from the effect of residual (e.g. Marangoni) flows in promoting a small degree of agglomeration there remains the question of whether the particles will be enveloped by the advancing front or "pushed" ahead of it. Analyses currently available of this phenomenon allow prediction of the critical (minimum) growth velocity for entrapment." (1, p. 55)

This TEXUS 10 experiment was the third in a series of investigations designed by Caton and/or Goodhew et al. to study the stability of a metallic dispersion (see Caton, TEXUS 7, Spacelab 1 (Chapter 17)). The specific objectives of the experiment were to (1) directionally solidify an aluminum melt containing a fine dispersion of insoluble particles, and (2) acquire "...data relevant to further exploration of the [above described] envelopment phenomenon." (1, p. 55)

During the mission, specimens of Al-Zr, Al-Nb, and Al-alumina fibre were to melted and solidified in the Swedish Space Corporation (SSC) furnaces. It was reported, however, that "...the furnaces malfunctioned and none of the specimens were processed correctly." (1, p. 55)

The experiment was reflown on TEXUS 12, (see Goodhew, TEXUS 12 (this chapter)).

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Metallic Matrix, Fibers, Fiber Dispersions, Dispersion Strengthening, Particle Dispersion, Particle Distribution, Particle Motion, Particle Coarsening, Particle Coalescence, Particle Agglomeration, Collisions, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Convection, Sedimentation, Marangoni Force, Interface Physics, Interfacial Energy, Inclusion and/or Rejection of Particles, Solidification Front Physics, Growth Rate, Furnace Malfunction, Incomplete Sample Processing

Number of Samples: It appears that a total of three samples were to be processed during the flight.
Sample Materials: : Al-Zr, Al-Nb, and Al-alumina fiber (Al*Zr*, Al*Nb*, Al*Al*O*)
Container Materials: unknown

Experiment/Material Applications:
Applications of this experiment are detailed in the **Experiment** section (above).

See also Caton, Spacelab 1 (Chapter 17) for a more general discussion.

References/Applicable Publications:

(1) Goodhew, P. J. and Clyne, D.: Retention of a Fine Precipitate Dispersion. In BMFT/DFVLR TEXUS 11/12 Abschlussbericht, 1985, p. 55. (discusses overall objectives and TEXUS 10 results; post-flight)

(2) Input received from Experiment Investigator, November 1989.

(3) Input received from Co-Investigator T. W. Clyne, July 1993.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 11
Launch Date/Expt. Date: April 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: Isothermal Heating Facility (IHF) furnace with cooling base (module used since TEXUS 1 but now with modifications to furnace design)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Metal Oxide Copper-Alumina (Cu-Al_2O_3) Suspension

This TEXUS 11 experiment was the twelfth in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, Spacelab 1, TEXUS 9 (this chapter); Pötschke, TEXUS 4 (two experiments), TEXUS 6, TEXUS 7 (all in Chapter 4)).

The specific objective of this experiment was to investigate the interaction of very small solid particles (from 0.05 to 0.5 micron) with the solidification interface. (Theoretical calculations had indicated that at the solidification velocities expected (0.02 to 0.1 cm/sec), the particles would be moved by the solidification front.)

Before the flight, a Cu-0.2 Al_2O_3 sample was prepared using wet mixing, vacuum degassing, sintering, and extrusion techniques. The sample was contained in a alumina crucible and placed in the TEXUS Experiment Module TEM 01-1 Furnace. The module was equipped with a cooling base. Just prior to lift-off, the sample was heated to 800 ^{o}C. During the low-gravity period of the flight, the sample was "almost completely molten" and directionally resolidified under a temperature gradient of 60 ^{o}C/cm.

Post-flight examination of the sample revealed the presence of gas bubbles (13% foam) which had been generated by the formation of CO. The inside of these bubbles were coated with alumina particles. The Cu matrix contained no particles. It was reported,

therefore, that the original objective of the experiment could not be realized. However, it was determined that convection caused the particles to reach the bubble walls. The alumina particles (poorly wetted by the melt) were rejected by the Cu and formed a film on the inside of the bubbles. This formation stabilized the bubbles. The lined bubbles coagulated and agglomerated due to Ostwald friction. Very small bubbles (<0.1 micron) were shifted by the solidification front.

"The coalesced bubbles could still be observed as their alumina cover had often conserved their initial shape. But there are only indirect indications of the effects of Ostwald ripening and the motion of the smaller bubbles: no pores are significantly smaller than 3öm. However, the alumina cover remaining from the bubbles which had disappeared was visible on the longitudinal section of the sample. According to theoretical calculations, this could result from Ostwald ripening of gas bubbles." (3, p. 332)

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Motion, Particle Wetting, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Convection, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Solidification Rate, Composites with Gases, Gas Formation, Bubbles, Bubble Formation, Bubble Stability, Bubble Coalescence, Particle Agglomeration, Coagulation, Ostwald Ripening, Pores, Foams, Thin Films

Number of Samples: one
Sample Materials: copper-alumina, Cu-0.2 Vol.% alumina (Cu*Al*O*)
Container Materials: clay crucible

Experiment/Material Applications:
See Neuschütz, TEXUS 1 (this chapter).

References/Applicable Publications:

(1) Pötschke, J.: Kupfer-Tonerde-Suspension. In TEXUS 11/12 Abschlussbericht, 1985, pp. 24-25. (in German; post-flight)

(2) Input received from Principal Investigator, J. Pötschke, September 1989 and August 1993.

(3) Gas Bubbles in a Copper-Alumina Suspension. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 332-333. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Unclear, either TEXUS Experiment Module TEM 01-2 or TEXUS Experiment Module TEM 01-1: Isothermal Heating Facility (IHF) with cooling base (module used since TEXUS 1 but now with modifications to furnace design)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
WC-Co Liquid Metal Suspension

This TEXUS 12 experiment was the thirteenth in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, Spacelab 1, TEXUS 9 (this chapter); Pötschke, TEXUS 4 (two experiments), TEXUS 6, TEXUS 7 (Chapter 4), TEXUS 11 (this chapter)).

The specific objective of this experiment was to investigate the interaction of very small particles (from 0.05 to 0.5 micron) with the solidification interface.

Reportedly, a single sample comprised of Co-35 wt.% WC was processed. <Note: A document which specifically discussed the inflight experimental procedure of this sample could not be located. However, the procedure may have been similar to that reported under Pötschke, TEXUS 11 ($CuAl_2O_3$ sample).>

The only information which appears to be available that discusses the post-flight analysis was the following: the Co-WC sample had "too much gas formation--no result." (2)

<Note: It is not clear if this Co-35 wt.% WC sample was the same as one of W. Graf's three Co-35 wt.% WC samples processed on TEXUS 12 (see Graf, TEXUS 12 (Chapter 13)). Graf also reported excessive gas formation.>

Key Words: Composites with Solid Particles, Binary Systems, Melt and Solidification, Particle Dispersion, Particle Distribution, Particle Motion, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Interface Physics, Solidification Front Physics, Inclusion and/or Rejection of Particles, Composites with Gases, Gas Formation, Bubbles, Bubble Formation

Number of Samples: one
Sample Materials: cobalt-tungsten carbide, Co-35 wt.% WC (W*C*Co*)
Container Materials: clay crucible

Experiment/Material Applications:
See Neuschütz, TEXUS 1 (this chapter).

References/Applicable Publications:
(1) Pötschke, J.: Kupfer-Tonerde-Suspension. In TEXUS 11/12 Abschlussbericht 1985, pp. 24-25. (in German; post-flight)

(2) Input received from Principal Investigator, J. Pötschke, September 1989 and August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility with Gradient Device (IHF/G)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
The Behavior of Aluminum Oxide Particles at the Solidification Front of Copper (WL-IHF 02)

This Spacelab D1 experiment was the fourteenth in a series of investigations designed by Neuschütz and/or Pötschke et al. to study the low-gravity solidification of metallic composites (see Neuschütz, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, Spacelab 1, TEXUS 9 (this chapter); Pötschke, TEXUS 4 (two experiments), TEXUS 6, TEXUS 7 (Chapter 4), TEXUS 11, TEXUS 12 (this chapter). The specific objectives of the D1 experiment were to (1) investigate the influence of the melting process and fluid state on the distribution of suspended particles within a composite and (2) examine the interaction between the suspended particles and moving solidification front.

Prior to the mission, two 150-mm long, 6-mm diameter samples were prepared: (1) Cu-2 vol.% alumina particles (1 to 20 micron particle diameter) and (2) Cu-2 vol.% Mo particles (1 to 4 micron particle diameter). The samples were contained within an alumina crucible protected by a tantalum cartridge.

During the mission, melting and directional solidification of the samples were performed using the Isothermal Heating Facility "with Gradient device" (IHF/G). (The various furnace translation rates were presented in Reference (5).)

Post-flight examination of the thermal data and processed samples indicated that (1) the Cu-alumina sample was exposed to a thermal gradient of 120 K/cm (planar solidification front) and (2) the

Cu-Mo sample was exposed to a thermal gradient of 45 K/cm (cellular solidification front). During the first 15 minutes of the experiment, the pulling rate unexpectedly varied from 0.025 to 0.1 mm/min. This anomaly "...resulted by a failure in the furnace control system. Here a holding time (v=0) was planned to stabilize the system thermally. Hence, the rate showed a fluctuation...." (5, p. 306)

X-ray examination of the processed samples indicated that portions of the samples "...contain[ed] pores [bubbles] causing convection and Marangoni effects. So only the first two solidification rates could be examined with respect to the pushing of particles." (5, p. 306) Examination of the suspension distribution illustrated that the particles had been pushed during solidification. In both samples the particles were shifted normal to the solidification interface.

Reportedly, the Cu-alumina sample contained an agglomeration of particles. This result was attributed to (1) the pushing of the particles by the planar solidification front and (2) the stringing of particles by the sample preparation procedure (hot extrusion). Examination of the Cu-Mo sample (cellular solidification) indicated a pearl-string distribution of the particles. This alignment was attributed to the pushing of the particles into the intercellular regions.

Reportedly, the observations and obtained values for solidification velocity corresponded reasonably well with theoretical determinations (see Reference (5)).

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Directional Solidification, Metallic Matrix, Metallic Oxides, Particle Dispersion, Particle Distribution, Particle Motion, Particle Agglomeration, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Convection, Marangoni Convection, Interface Physics, Solidification Front Physics, Planar Solidification Interface, Cellular Characteristics, Inclusion and/or Rejection of Particles, Thermal Gradient, Translation Rate, Solidification Rate, Composites with Gases, Gas Formation, Bubbles, Bubble Formation, Pores, Furnace Malfunction

Number of Samples: two
Sample Materials: (1) Cu-2 vol.% alumina particles; (2) Cu-2 vol.% Mo particles
(Cu*Al*O*, Cu*Mo*)
Container Material: alumina
(Al*O*)

Experiment/Material Applications:
See Neuschütz, TEXUS 1 (this chapter).

References/Applicable Publications:
(1) Pötschke, J.: The Behavior of Aluminum Oxide Particles at the Solidification Front of Copper. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 133-134. (preflight)

(2) Input received from Principal Investigator J. Pötschke, September 1989 and August 1993.

(3) Pötschke, J. and Rogge, V.: Das Verhalten suspendierter Teilchen an der Erstarrungsfront von Kupfer. Natur wissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 381-383. (in German; post-flight)

(4) Pötschke, J. and Rogge, V.: Das Verhalten supendierter Teilchen an der Estarrungsfront von Kupfer. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 74-77. (in German; post-flight)

(5) Pötschke, J. and Rogge, V.: The Behavior of Suspended Particles at the Solidification Front of Copper. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 304-308. (post-flight)

(6) Pötschke, J. and Rogge, V.: On the Behaviour of Foreign Particles at an Advancing Solid-Liquid Interface. Journal of Crystal Growth, Vol. 94 (1989), pp. 726-738. (theoretical treatment)

(7) Pötschke, J.: Werkstoff-Experimente im Weltraum unter Schwerelosigkeit. Technische Mitteilungen Krupp 1/1986, pp. 21-24. (in German)

(8) Sprenger, H. J., Pötschke, J., Potard, C., and Rogge, V.: Composites. In Fluid Sciences and Materials Science in Space, H. U. Walter, ed., Springer-Verlag, Berlin, 1987, pp. 567-597. (related topics)

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Experiment Origin: Great Britain
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Swedish Space Corporation (SSC) TEXUS experiment module furnaces (the module had been upgraded after TEXUS 10)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Retention of a Fine Precipitate Dispersion

This experiment was the fourth in a series of investigations designed by Goodhew and/or Caton et al. to study the stability of a metallic dispersion (see Caton, TEXUS 7, Spacelab 1 (Chapter 17), TEXUS 10 (this chapter)). The specific objectives of the experiment were the same as those detailed under Caton, TEXUS 10: (1) to directionally solidify an aluminum melt containing a fine dispersion of insoluble particles, and (2) to acquire "...data relevant to further exploration of the [particle] envelopment phenomenon." (1, p. 55)

During the mission, it appears that specimens of Al-Zr, Al-Nb, and Al-alumina fibre were melted and solidified within the Swedish Space Corporation (SSC) furnaces. Reportedly, however, "Although Extensive ground tests were undertaken, both at Surrey on a model test and at the Swedish Space Corporation (SSC/ACR), and many specimens were processed without incident, the cartridges all leaked during the actual flight and again none were processed correctly. Therefore no results were gained...." (1, p. 55)

No further information concerning the TEXUS 12 experiment could be located at this time.

The experiment was reflown on TEXUS 14a (see Goodhew, TEXUS 14a (this chapter)).

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Metallic Matrix, Fibers, Fiber Dispersions, Dispersion Strengthening, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Inclusion and/or Rejection of Particles, Solidification Front Physics, Liquid Leakage, Processing Difficulties

Number of Samples: three
Sample Materials: Unknown; it appears that they were: Al-Zr, Al-Nb, and Al-alumina fiber.
(Al*Zr*, Al*Nb*, Al*Al*O*)
Container Materials: unknown

Experiment/Material Applications:
See Caton, TEXUS 10 (this chapter) or Caton, Spacelab 1 (Chapter 17).

References/Applicable Publications:
(1) Goodhew, P. J. and Clyne, D.: Retention of a Fine Precipitate Dispersion. In BMFT/DFVLR TEXUS 11/12 Abschlussbericht, 1985, p. 55. (post-flight)

(2) Experiment-Modul SSC. In BMFT/DFVLR TEXUS 11/12 Abschlussbericht, 1985, p. 52. (in German; experiment module)

(3) Input received from Experiment Investigator, November 1989.

(4) Input received from Co-Investigator T. W. Clyne, July 1993.

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Experiment Origin: Great Britain
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation TEXUS Experiment Module: Gradient Furnace Assembly (GFA) Module designed for directional solidification experiments (employed originally on TEXUS 12)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Retention of a Fine Precipitate Dispersion

This TEXUS 14a sounding rocket experiment was the fifth in a series of investigations designed by Goodhew and/or Caton et al. to study the stability of a metallic dispersion (see Caton, TEXUS 7, Spacelab 1 (Chapter 17), TEXUS 10 (this chapter); Goodhew, TEXUS 12 (this chapter)).

A description of the specific experimental objectives or equipment setup could not be located.

Reportedly, due to an unexpected "wobbling motion" of the TEXUS rocket, uncontrollable accelerations were produced on the vehicle and the desired low gravity of 10^{-4} g was not attained. The investigator briefly mentioned that the experimental results were useless.

Documentation detailing any further results of TEXUS 14a does not appear to be available.

The experiment was reflown on TEXUS 14b (see Goodhew, TEXUS 14b (this chapter)).

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Metallic Matrix, Fibers, Fiber Dispersions, Dispersion Strengthening, Particle Dispersion, Particle Distribution, Particle Motion, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Inclusion and/or Rejection of Particles, Solidification Front Physics, Growth Rate, Rocket Motion, Acceleration Effects

Number of Samples: unknown
Sample Materials: unknown, possibly Al-Zr, Al-Nb, and Al-alumina fiber.
(Al*Zr*, Al*Nb*, Al*Al*O*)
Container Materials: Unknown, possibly boron nitride
(B*N*)

Experiment/Material Applications:
Please refer to Caton, TEXUS 10 (this chapter) or Caton, Spacelab 1 (Chapter 17).

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 53-55. (in German; post-flight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 60-61. (gradient furnace description)

(3) Input received from Experiment Investigator, November 1989.

(4) Input received from Co-Investigator T. W. Clyne, July 1993.

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Experiment Origin: Great Britain
Mission: TEXUS 14b
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation TEXUS Experiment Module: Gradient Furnace Assembly (GFA) Module designed for directional solidification experiments (employed originally on TEXUS 12)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Retention of a Fine Precipitate Dispersion

On Earth, heating of an aluminum melt containing insoluble fibers above the melting point of the matrix material generally results in the rapid sedimentation and agglomeration of the particles.

This TEXUS 14b sounding rocket experiment was the sixth in a series of investigations designed by Goodhew and/or Caton et al. to study the stability of a metallic dispersion (see Caton, TEXUS 7, Spacelab 1 (Chapter 17), TEXUS 10 (this chapter); Goodhew, TEXUS 12, TEXUS 14a (this chapter)). The specific objectives of the experiment were to (1) verify the theory concerning particle engulfment published by Chernov et al. [Sov. Phys. Crystallog., Vol. 21, 369 (1976)] and (2) "...directionally solidify...an aluminum melt containing insoluble alumina fibres." (1, p. 62)

"Chernov... predicts that, for a spherical particle, engulfment will cease and particle pushing begin when the velocity of the solidifying material drops below a critical value v_n...." (1, p. 65) This critical velocity, v_n, is a function of (1) a constant B (which defines the fall in chemical potential in a thin film compared with the bulk liquid), (2) the surface tension, (3) the dynamic viscosity, and (4) a term R. <Note: The term R, which was undefined in Reference (1), is presumably the particle radius.>

Prior to the mission, three 5.4 mm-diameter, 25 mm-long samples of pure aluminum-"Saffil" alumina fibers (4-5 vol.%) were prepared. Reportedly, the fibers had a diameter of 2-3 μm "...which in small aspect ratio fibres is predicted to exhibit engulfment breakdown and particle pushing at solidification rates available in sounding rocket experiments." (1, p. 62)

During the mission, the samples were processed within the Gradient Furnace Assembly (GFA) Module. Differences in sample heat sink design made it possible to generate a different heat extraction rate for each sample. "The thermal history of each sample was recorded by three thermocouples and the solidification front velocity calculated." (1, p. 62)

While sample cartridges employed during TEXUS 12 had leaked (see Goodhew, TEXUS 12), post-flight inspection of the directionally solidified TEXUS 14b samples indicated that no such leakage occurred during this experiment.

<Note: The discussion of the post-flight analysis as presented in Reference (1) was somewhat difficult to follow. It is detailed in the following paragraphs, almost in its entirety.>

Reportedly, "The final system geometry, necessary to obtain different heat extraction rates, makes accurate heat flow modelling highly complex. However, it seems that in practise[sic] the solidification front rapidly entered a quasi-steady state regime. It has therefore been assumed that the growth front velocities were constant in each case and these have been estimated from the thermocouple traces. These traces can be interpreted in conjunction with the final solidification structure observed in an etched sample in which it is observed that the aluminum matrix has solidified with a dendritic morphology." (1, p. 63) Thus, the first flight sample was solidified at a slow rate (growth front velocity 0.29 mm/s), the second sample at a medium rate (growth front velocity 0.35 mm/s) and the third sample at a fast rate (growth front velocity 1.0 mm/s).

The three flight samples were compared to ground-based samples solidified at a "slow" rate (growth front velocity 0.46 mm/s), a medium rate (growth front velocity 0.46 mm/s) and a fast rate (growth front velocity 1.14 mm/s). <Note: It is not clear why both the slow and medium growth rates for the ground-based samples were reported to be the same rate.> "Three sections were machined from each sample (5, 10, and 15 mm from the heatsink and polished." (1, p. 63)

Reportedly, both slowly-solidified, low-gravity and ground-processed samples exhibited a "...significant increase in the number of sub-4μm^2 particles from the bottom to the top of the

specimen. These particles seem to have been pushed of [sic] the solidifying dendrites reducing the number at the bottom and increasing the number at the top..." (1, p. 63-65.)

"In the fast solidification rate specimens the pattern is not so clear. In the space processed specimen the sub-4μm^2 particles start at the average concentration then decrease in the middle and increase at the top. It is possible that the assumption of constant dendrite velocity throughout solidification is not valid and that in this case the velocity started above the critical velocity and then dropped below it, starting to push particles in the middle of the specimen.

"In the fast (ground processed) specimen which has the highest average dendrite velocity there is little statistically significant particle redistribution." (1, p. 65)

<Note: Results detailing particle redistributions in the low-g and Earth-processed specimens solidified at a medium rate were not presented in Reference (1).> Histograms of particle distributions in the slowly solidified and fast solidified flight and space-based samples were presented in Reference (1).

"Comparison of the calculated values for the critical velocity for the smallest particles, with the average dendrite velocities achieved in this experiment, suggests particle pushing should occur in only the slow and medium solidification rate specimens. This is confirmed in the particle distribution histograms... although theory predicts a critical velocity approximately three times slower than that seen in the experiment. However in view of the uncertainty in the value of constant B and the surface tension the agreement is reasonable." (1, p. 65)

The investigator reported that because it was difficult to vary the solidification rates widely, the results were rather inconclusive but did not contradict the theory.

Key Words: Composites with Solid Particles, Dispersion Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Metallic Matrix, Fibers, Fiber Dispersions, Dispersion Strengthening, Particle Dispersion, Particle Distribution, Particle Motion, Particle Agglomeration, Collisions, Solid/Liquid Dispersion, Solid/Liquid Interface, Homogeneous Dispersion, Stability of Dispersions, Viscosity, Sedimentation, Interface Physics, Surface Tension, Interfacial Energy, Inclusion and/or Rejection of Particles, Solidification

Front Physics, Growth Rate, Solidification Rate, Dendrites, Dendritic Structure

Number of Samples: three
Sample Materials: aluminum-"Saffil" alumina fiber system (Al*)
Container Materials: boron nitride (B*N*O*)

Experiment/Material Applications:
Space processing eliminates sedimentation-led coarsening mechanisms and enables the investigation of the engulfment/pushing phenomena.

See also Caton, TEXUS 10 (this chapter) or Caton, Spacelab 1 (Chapter 17).

References/Applicable Publications:
(1) Adkins, N.J.E. and Goodhew, P. J.: Solidification of Fine Dispersions in Microgravity. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 62-65. (postflight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 60-61. (gradient furnace description)

(3) Input received from Experiment Investigator, November 1989.

(4) Input received from Co-Investigator T. W. Clyne, July 1993.

(5) Solidification of Fine Dispersions in Microgravity. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, SP-1132, February 1991, pp. 306-307. (postflight)

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Principal Investigator(s): Langbein, D. (1)
Co-Investigator(s): Roth, U. (2)
Affiliation(s): (1,2) Battelle-Institute, Frankfurt, Germany

Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA, Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) Furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Particles at Melting and Solidification Fronts (WL-IHF 06)

"Microgravity offers the chance of producing compact monocrystalline composites by melting and solidification of uniform mixtures of a metal and solid particles precondensed on [the] ground. The absence of buoyancy prevents the rise and sinking of the incorporated particles in the melt, the absence of thermal convection reduces the probability for the particles to contact each other and to coagulate or to deposite [sic] at the crucible wall. Whereas deposition at the wall may be affected by the mutual wetting properties of [the] melt, particles and wall, particle coagulation lowers their interface energy in all cases....

"During such investigations under microgravity, a free surface of the melt with a gas volume must be carefully avoided. Such a surface favours particle deposition and coagulation, on the one hand. It gives rise to capillary effects and to interface convection, on the other hand. A free surface may be excluded by means of a volume compensation system, which balances for the volume expansion of the sample during heating and melting and for the volume shrinking during cooling and solidification." (4, p. 309)

This Spacelab D1 experiment was designed to examine the low-gravity behavior of particles displaced during melting and solidification. The specific objective of the research was to study (at various solid/liquid interface velocities) the repulsion of particles by (1) the melting front and (2) the solidification front.

Prior to the D1 mission, a copper sample containing 1 vol.% molybdenum particles (2 to 4 microns in diameter) was placed in an alumina container (0.5 mm wall thickness). Directly above

the sample (within the alumina container) was a section of electrolytic copper. Directly above the electrolytic copper was a capillary volume compensation system (also within the container). The entire arrangement was contained within a tantalum crucible.

The volume compensation system consisted of a cylinder of magnesium silicate (10 mm in length). Nineteen 1-mm diameter holes were drilled into the cylinder. The system was designed to avoid Marangoni convection in the melt, and thus reduce coagulation and skeleton formation of the particles. "Magnesium... [silicate] is not wetted by molten copper such that a convex meniscus in the holes and a positive capillary pressure in the melt results. The melt penetrates into the capillaries during heating and melting and is pressed back during cooling and solidification." (4, p. 310) The section of electrolytic copper (placed between the volume compensation system and the sample material) prevented penetration of molybdenum particles into the capillaries.

During the Spacelab D1 mission, the Isothermal Heating Facility (IHF) was used to melt and resolidify the sample. The intent was to directionally melt the sample from the top downward with an increasing traveling rate. This was to be accomplished by moving the chill block at different speeds during the melt process. Then, the sample was to be directionally solidified upward with a decreasing rate. This procedure would have permitted (at various rates of movement) the investigation of the interaction between the molybdenum particles and the (1) melting front and (2) solidification front. However, because of "...difficulties with the vacuum system of the IHF and further delay during conversion to the gradient mode.... The slow unidirectional melting had to be canceled." (4, p. 310) Instead, the sample was rapidly melted from its center and subsequently directionally solidified with decreasing traveling rates of the chill-block.

Post-flight removal of the sample from the tantalum cartridge was performed without difficulty. The outer surface of the sample exhibited bubbles "...in the upper part of the sample only, which rapidly solidified after switching off the IHF. Whereas the copper melt has wetted the alumina crucible, the solid phase obviously detached from it. The copper sample and the volume compensation system separated too, when the crucible was opened.... 19 pimples, the remainders of the menisci which were pushed out of the 19 capillaries of the volume compensation system are clearly visible.

"Due to melting of the sample starting at its centre rather than at the volume compensation system, copper penetrated through cracks in the alumina crucible and moved along the capillary slit between the two crucibles [tantalum and alumina]...." (4, pp.

310-311)

<Note: A detailed discussion of the sample is provided in Reference (4). The following is a brief summary of the X-ray image analysis and related discussion.> Post-flight examination of the sample revealed no characteristics of global convection within the melt. There was no clustering or skeletal formation of the molybdenum particles.

Several different types of bubbles were observed in the samples. Spherical bubbles, some of which contained copper, were present. Molybdenum particles were also found within the bubbles. Some of these particles were 10 to 20 microns in diameter and exhibited a perfect crystalline shape. This result indicated that the Mo particles grew within the bubbles. In addition to spherical bubbles, tear-shaped bubbles were also observed. The slim end of these bubbles pointed toward the hot end of the sample (an analysis of this result is included in Reference (4)).

Reportedly, "The volume percentage of bubbles clearly increases... with increasing sample temperature and time. The number of bubbles decreases in the same direction, e.g., with increasing volume content of the bubbles[,] coagulation was more frequent. This is strengthened by the fact that in the vicinity of large bubbles only very few small bubbles are found." (4, p. 312)

"If a uniform gas generation in the molten sample is assumed, the increase in volume percentage of the bubbles from bottom to top must be attributed to Marangoni migration. Alternatively, an increase in gas generation with increasing duration in the molten state and the maximum melt temperature may be considered. The latter assumption is supported by the absence of bubbles in the upper sample region consisting of electrolyte copper. In this region no oxygen for bubble formation was available. A further argument against Marangoni migration of the bubbles is the strong covering of their surfaces by molybdenum particles. These did not just deposite[sic] but also crystallized within the bubbles, which requires long times free of stirring." (4, p. 313) Additional discussion of the possible bubble migration modes are presented in Reference (4).

It was concluded that the volume compensation system did not work as intended. The system "...was hardly able to do so, since melting did not start at its position. It appears that... [it] worked more due to its porosity than due to the intended capillary mechanism. In follow-up experiments under microgravity conical capillaries should be used, in order for accidental differences in diameter and thus in capillary pressure not to cause successive rather than simultaneous filling and clearing of the

capillaries." (4, p. 314)

<Note: Langbein also performed solidification studies with immiscible materials in conjunction with Heide (see Heide, TEXUS 5, TEXUS 7, TEXUS 8, TEXUS 9 (Chapter 17)). Also of interest is Langbein, D1 "Mixing and Demixing of Transparent Liquids" (Chapter 17).>

Key Words: Composites with Solid Particles, Binary Systems, Dispersion Alloys, Melt and Solidification, Directional Solidification, Metallic Matrix, Particle Dispersion, Particle Distribution, Particle Motion, Particle Wetting, Coagulation, Suspension of Particles, Stability of Dispersions, Stability of Suspensions, Solid/Liquid Dispersion, Solid/Liquid/Gas Dispersion, Solid/Liquid Interface, Sedimentation, Segregation, Convection, Marangoni Convection, Marangoni Movement (Migration) of Bubbles, Interface Physics, Interfacial Energy, Buoyancy Forces, Wetting, Wetting of Container, Solidification Front Physics, Inclusion and/or Rejection of Particles, Thermal Gradient, Solidification Rate, Composites with Gases, Gas Formation, Bubbles, Bubble Formation, Porosity, Crucible Effects, Free Surface Elimination, Meniscus Shape, Volume Compensation, Volume Expansion, Capillaries, Capillary Forces, Liquid Leakage, Hardware Malfunction, Processing Difficulties

Number of Samples: one
Sample Materials: copper containing 1 vol% molybdenum particles (2 to 4 microns in diameter)
(Cu*Mo*)
Container Materials: alumina (0.5 mm wall thickness) within a tantalum crucible
(Al*O*, Ta*)

Experiment/Material Applications:
A copper sample with molybdenum particles was used for this experiment because the sample was to be compared to specific materials processed during earlier microgravity studies (see Deruyttere, TEXUS 9; Pötschke, TEXUS 11 (Chapter 17)) and another Spacelab D1 investigation (see Pötschke, Spacelab D1 (Chapter 17)).

References/Applicable Publications:

(1) Langbein, D. and Roth, U.: Wirkungen eines kapillaren Volumenausgleichs beim Schmelzen und Estarren unter Mikrogravitation. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 106-110. (abstract only; in German)

(2) Langbein, D. and Pötschke, J.: Particles at Melting and Solidification Fronts. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 135-136. (preflight)

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(5) Input received from Principal Investigator D. Langbein, August 1993.

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CHAPTER 6

CONTAINERLESS PROCESSING

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Affiliation(s): (1) During Skylab: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama, Currently: Deceased; (2) Grumman Aerospace Corporation, Bethpage, New York

Experiment Origin: USA
Mission: Skylab, SL-2, First Skylab Manned Mission
Launch Date/Expt. Date: June 1973 (month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) (located forward from the Multiple Docking Apparatus (MDA) area in the Skylab manned environment)
Processing Facility: Sphere forming assembly (electron beam welder) with two sample specimen wheels (FO1 and FO2) and sphere catchers
Builder of Processing Facility: Unknown

Experiment:
Sphere Forming (M553)

This Skylab SL-2 experiment was designed to study the low-gravity containerless solidification of materials with a face-centered cubic structure. The specific objective of the experiment was to demonstrate the effects of reduced gravity and space vacuum on (1) pure nickel (solidified with an undercooling not possible on Earth), (2) a Ni-12 wt.% Sn alloy (which has a wide freezing range), (3) a Ni-1 wt.% Ag alloy (which (a) has a narrow freezing range and (b) cores on solidification), and (4) a Ni-30 wt.% Cu alloy (which has a wide freezing range but which has constituents of near equal density).

The "M512 Furnace Facility" was used to process the materials. The facility consisted of (1) a vacuum chamber, (2) an electron beam gun, (3) a sample wheel holder, (4) an indexing motor, (5) a 16 mm film camera, and (6) a sphere catcher.

Each sample wheel had 14 spokes dedicated to holding samples. A single sample was placed on the end of each of these spokes. The indexing motor was used to turn the wheel and thus position each sample in the path of the electron beam for melting. A fifteenth spoke (in each of the sample wheels) was used to align the beam. The camera recorded the processing of each sample.

Each wheel held four pure Ni samples, four Ni-1 wt.% Ag samples, four Ni-12 wt.% Sn samples, and two Ni-30 wt.% Cu samples. Three of the samples on each wheel were attached to metal rods and

remained fixed during melting and resolidification. The remaining 11 samples were mounted to ceramic pedestals by a metallic sting. Once the sample melted, the sting was retracted to allow solidification of the sample while floating free. The retraction of the sting also automatically shut off the electron beam. The 11 free-floating samples were collected in a container using a vacuum system.

During processing of the samples on the first wheel, "The crewman reported difficulty in aligning the electron beam on the target, and the pressure level in the chamber degraded while the electron beam gun was operating. On the fourth sample, the gun automatically turned off after 1 second of the normal 5-second melt time. When the pressure level approached 1×10^{-4} torr, the gun was turned off and the experiment delayed until the pressure decreased to 1×10^{-5} torr.... The crewman reported that some samples on the first wheel had not completely melted." (10, p. 12-44)

Difficulties were also encountered during the processing of the second wheel samples. "At times the electron beam gun could not be turned off except by opening the battery main circuit breaker. In addition, there was occasionally a blue glow in the work chamber. After consultation, it was decided to terminate operation of the experiment after the next normal turnoff of the electron beam gun. Seven of the 14 samples were completed before experiment termination....

"The decision to terminate the experiment early was based on the limited time remaining to perform... [another experiment]... and the increased crew time being required to perform... [this] sphere forming experiment. Outgassing of the samples during the electron beam gun operation caused the pressure in the chamber to increase. The increased pressure was causing a high voltage discharge in the chamber, which accounts for the blue glow.... The cause of alignment problems on the first specimen [wheel] is unknown. For some reason the 16-millimeter film coverage for that specimen [wheel] did not start until the third sample.

"The film for the second specimen [wheel] shows an excellent alignment of the gun and the target. The gun turned off after 1 second on the fourth sample of specimen wheel 1 because the electron beam struck the ceramic post and probably melted the sting. The films taken during that operation verify that the beam struck the post, and melting of the sting will turn the gun off." (10, p. 12-44) It was also reported that all samples on wheel 1 were only partially melted.

Post-flight examination of the camera film revealed that none of the free-floating samples solidified prior to contacting the container wall. All of the low-gravity specimens exhibited a sphericity value (R_{max}/R_{min}) of typically 1.01 to 1.04 (compared to 1.28 for similarly processed 1-g samples). The lower portion of the samples (both 1-g and low-g) were composed of three sections: (1) the bottom section was composed of heterogeneous or localized nucleation at the ceramic pedestal base of unmelted solid adjacent to the base, (2) the middle section was composed of epitaxially nucleated dendrites which grew rapidly into the melt, and (3) the top section was a region of nucleation and growth that occurred at the surface. The dendritic region was the last to solidify and "As the ratio of thermal gradient (G) to growth rate (R) decreased to a critical level, the columnar growth decayed to an equiaxed free dendritic form...." (1, p. 103) The relative size of these regions varied depending upon the thermal history of the samples. Also, platelet, cellular, and dendritic growth mechanisms could have been active in the alloys which solidified by a surface nucleation mechanism. Microcompositional mapping (<100> directions) indicated that the growth mechanism was determined by G and R rather than compositional fluctuations.

It was expected that "...the differing alloy content would affect the degree of constitutional supercooling during the bivariant portion of the solidification and would, in concert with the thermal parameters, result in a diversity of the solidification mechanisms." (1, p. 104) It was reported that this expectation was confirmed. The Ni-1 wt.% Ag alloy primarily solidified with a cellular mechanism while the nickel-tin and nickel copper alloys solidified dendritically. The solidification mechanisms discussed in the previous paragraph only occurred sequentially as surface reactions when G/R was high. In the bulk, where G/R decreased, either cellular (silver alloy) or dendritic (tin and copper alloys) solidification occurred.

Examination of the eutectic solidification in the Ni-12 wt.% Sn sample revealed a terraced morphology with Ni_3Sn intermetallic decorating the proeutectic dendrite tips. The platelet and cellular solidification regions also exhibited a terraced structure. Neither the terraced structure nor the intermetallic decoration of the dendrite tips could be located in similarly processed ground samples indicating that these characteristics may be enhanced by reduced convection.

Internal voids were located in Ni-1 wt.% Ag samples and to a lesser degree in some of the Ni-12 wt.% Sn samples. The volume of the voids was such that they were not due to solidification shrinkage. Microchemical analysis of the void surfaces indicated that they were due to metallic gas evolution during processing.

Analysis indicated that these voids may have resulted from a decrease in hydrostatic pressure head within the molten liquid. This reduction of the pressure head was caused by the reduced gravity and allowed low-pressure phase reactions to become bulk reactions rather than surface reactions.

Key Words: Containerless Processing, Spheres, Sphericity, Binary Systems, Eutectics, Intermetallics, Surface Tension, Melt and Solidification, Electron Beam Melting, Undercooling, Supercooling, Thermal Gradient, Growth Rate, Solid/Liquid Interface, Liquid/Gas Interface, Drops, Cellular Morphology, Platelet Habit, Dendritic Solidification, Dendrites, Composition Distribution, Surface Morphology, Collisions, Outgassing, Voids, Gas Formation, Nucleation, Hydrostatic Pressure, Space Vacuum, Direct Exposure to Space Environment, Incomplete Sample Processing, Hardware Malfunction, Photographic Difficulties,

Number of Samples: twenty-eight
Sample Materials: (1) pure nickel, (2) nickel-12% tin alloys with wide freezing range, (3) nickel-1% silver alloys with with narrow melting range, and (4) nickel-30% copper alloys with wide melting range
(Ni*, Ni*Sn*, Ni*Ag*, Ni*Cu*)
Container Materials: not applicable

Experiment/Material Applications:
Nickel and nickel alloys were selected for this experiment because (1) they solidify in a face-centered cubic structure and (2) the solidification theory for these types of materials has been extensively studied.

Also see Experiment section (above).

References/Applicable Publications:
(1) Larson, D. J.: Skylab M553 Sphere Forming Experiment. In Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. 1, April 30-May 1, 1974, Marshall Space Flight Center, Alabama, June 1974, pp. 101-113. (post-flight results)

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(9) Chassay, R. P. and Schwaniger, A.: Low G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

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Co-Investigator(s): Unknown
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Experiment Origin: USA/Federal Republic of Germany
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt Date: September 1973 (month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab manned environment
Processing Facility: Multipurpose Electric Furnace System (MEFS) M-518
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Growth of Spherical Crystals (M-560)

This Skylab experiment was the first in a series of investigations designed by Walter to study the directional solidification of a quasi-containerless melt. The objectives of the investigation included: (1) determining the feasibility of low-gravity (a) containerless processing of single crystals and (b) production of homogeneously doped, structurally perfect semiconductor materials, and (2) correlating the resulting physical properties of the space crystals to (a) similarly processed samples grown on Earth, and (b) theoretical values for ideal crystals.

Three indium antimonide (InSb) crystals were configured in the Skylab Multipurpose Electric Furnace System (M-518). Each of the InSb samples was directionally solidified in a heating chamber (length = 25 cm, i.d. = 2.14 cm) which had three temperature zones: (1) a 5-cm hot zone with resistance heater and graphite leveler, (2) a 6.3-cm gradient zone, and (3) a 14-cm heat extraction zone.

All three samples were processed using identical procedures: "An oriented [111 direction] cylindrical single crystal of InSb (8.0 mm dia., 48 mm long) was mounted [at one end] into a graphite base that was located in the heat extraction section of the gradient furnace. From this support the crystal extended [free from the container walls] through the gradient section of the furnace into a hemispherical graphite cavity that is located in the heated section of the furnace. As the graphite cavity is heated, the crystal slowly melts, starting at the bottom of the

cavity. The melt adheres to the end of the seed crystal [in the form of a drop] and detaches from the graphite as melting continues." (10, p. 3) In all of the chambers "The drops were solidified by removing heat through the seed crystals while heat losses from the surfaces... [were] compensated. In this manner the crystalline material in contact with the seed grows inside the drop with the liquid on the drop surface being the last to solidify, thus eliminating mechanical strain due to volume change on solidifying." (5, p. 5-67) Approximately 1.6 cm of each crystal was remelted and solidified in space.

Since the melt was nearly the shape of a spherical drop, "...it was expected that the crystal would solidify into a spherical shape also, which is the reason the growth cavity was made with a hemispherical end cap.... Instead a teardrop shape developed. This shape is determined by the meniscus angle... and the volume change associated with solidification.... [A] peculiar tip was produced when the crystal, being longer than anticipated grew into the crucible wall." (3, p. 56)

Post-flight examination of the samples indicated that their surfaces, with the exception of small areas where small foreign particles had accumulated, were very smooth and reflective. All samples showed a distinct "ring-shaped groove" which probably resulted from a Skylab maneuver which occurred 52 minutes after sample cooldown was initiated. From this groove, average growth rates of the samples were determined to be 10.4 mm/hr, 12.8 mm/hr, and 13 mm/hr. The highly perfect single crystals showed lancet-shaped growth facets, which were of the {111}, {-1-1-1}, {110}, and {211} planes, with the {110} facets being extremely well developed followed by the {111} and {-1-1-1} facets. The crystals were somewhat elongated along the growth axis due, in part, to an approximately 13% volume increase. The elongation may also have been due to the fact that "The center portion coincides with the {110} planes, and the angle at which the crystal diameter initially decreases is close to the direction of B faces ((-111), (1-11), (11-1)). Since these are the type of facets that are largest and best developed, it may be concluded that shaping of the growing crystal was mainly due to these facets." (10, p. 5).

Chemical etching of wafers (from two of the space crystals), which had been cut along the {111} planes (perpendicular to the [111] growth direction), revealed indium edge dislocations on {-1-1-1} planes which terminated with indium atoms. A plot of dislocation density vs. sample length showed a maximum at the beginning of growth, followed by a linear decrease. The last solidified portion of the samples showed an increase in dislocation density. The density most likely increased during the last portion of the growth process because of the increase in radial

thermal gradients in this section of the sample. There is also a local maximum at the beginning of the last third of the crystal which coincides with the Skylab maneuver noted above. The average dislocation density was reported to be 100/cm^2.

Key Words: Containerless Processing, Semi-Containerless Melt, Melt and Solidification, Directional Solidification, Supercooling, Thermal Gradient, Binary Systems, Semiconductors, Single Crystals, Seed Crystals, Spherical Crystals, Dopant, Dopant Distribution, Sample Homogeneity, Drops, Spheres, Sphericity, Free Surface, Meniscus Shape, Buoyancy-Driven Convection, Growth Rate, Surface Morphology, Crystal Morphology, Dislocations, Strain, Sample Microstructure, Container Shape, Material Interaction with Containment Facility, Surface Tension, Solid/Liquid Interface, Liquid/Gas Interface, Volume Expansion, Volume Change, Contamination Source, Acceleration Effects

Number of Samples: three
Sample Materials: indium-antimonide (In*Sb*)
Container Materials: not applicable

Experiment/Material Applications:
Processes such as the preparation of single crystals and the purification of compounds (both by liquid-solid reactions) are often difficult to achieve because of limitations imposed by the crucible. Compatibility problems between sample and crucible materials such as temperature limitations, reactivity, and/or contamination, are difficult to overcome. If low-gravity, containerless processing of these materials could be realized crucible limitations would be nonexistent. Low-gravity processing would result in nearly complete elimination of gravity-driven convection, thus simplifying system modeling and solidification process control.

References/Applicable Publications:
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(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 22-24, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) Naumann, R. J. and Herring, H. W.: Experiment M560, Seeded, Containerless Solidification of Indium Antimonide. In Materials Processing in Space: Early Experiments, NASA SP-443, pp. 56-57. (post-flight)

(4) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

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Experiment Origin: USA/Federal Republic of Germany
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt Date: December 1973 (month during which experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab manned environment
Processing Facility: Multipurpose Electric Furnace System (MEFS) M-518
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Containerless Processing of Single Crystals

This Skylab experiment was the second in a series of investigations designed by Walter to study the directional solidification of a containerless melt (see Walter, Skylab SL-3).

During the mission, three samples of indium antimonide (InSb) were melted and directionally solidified using the Multi-Purpose Electric Furnace System (M-518). One of the samples was heavily doped with selenium in order to investigate variations in dopant concentration as a function of convection, mechanical and thermal instabilities, constitutional supercooling, etc. Reportedly, the experimental procedure was the same as that employed during Walter's earlier SL-3 experiment (see Walter, Skylab SL-3).

Post-flight analysis of the undoped space crystals confirmed the data obtained from the SL-3 mission samples. The crystals were tear-drop shaped; the {110} facets very well developed. The {111}, {-1-1-1}, and particularly the {211} facets were smaller and more disturbed than the {110} facets. Chemical etching showed edge dislocations on the {-1-1-1} planes which terminated with In atoms. The pattern of dislocation distribution within the crystal and the dislocation density ($100/cm^2$) were also similar to the SL-3 samples with the exception that (1) the SL-4 samples did not exhibit the ring-shaped mark (which reportedly had resulted during a vehicle maneuver during SL-3) or (2) the corresponding local increase in dislocation density.

The Se doped InSb sample was grown in approximately the {110} direction with a space-grown length of approximately 2 cm (1-cm diameter). The interface between the solid and liquid portions was convex throughout the solidification process. The dislocation density was approximately $100/cm^2$ in the first portion of the sample, but decreased to nearly zero in the last half. Homogeneous doping of the crystal occurred in the center of the sample whereas striations were seen in the top and bottom portions. Reportedly, three types of very distinct striations were observed: "Rotational striations in the [Earth-produced] Czochralzki-grown seed..., and in the space grown section off-facet striations... and striations associated with the formation of a peripheral (111)B solid-liquid interface facet...." (6, p. 44) The striations formed during the low-gravity processing could not be explained by existing theory; therefore, a new concept based on kinetic supercooling was proposed by Walter. Kinetic supercooling would explain not only the striations observed in this sample, but also account for certain non-rotational striations seen in Earth-grown crystals. Reportedly, evidence of fluid-flow effects and convective interference could not be found.

Key Words: Containerless Processing, Semi-Containerless Melt, Melt and Solidification, Directional Solidification, Supercooling, Thermal Gradient, Binary Systems, Ternary Systems, Semiconductors, Single Crystals, Seed Crystals, Spherical Crystals, Dopant, Dopant Distribution, Sample Homogeneity, Drops, Spheres, Sphericity, Meniscus Shape, Free Surface, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Surface Morphology, Dislocations, Concentration Distribution, Sample Microstructure, Container Shape, Surface Tension, Solid/Liquid Interface, Liquid/Gas Interface, Interface Shapes, Striations, Rotational Striations

Number of Samples: three
Sample Materials: Samples 1 and 2: indium-antimonide; Sample 3: indium-antimonide doped with selenium (concentration approximately 10^{19} atoms of Se/cc)
(In*Sb*, In*Sb*Se*)
Container Materials: not applicable

Experiment/Material Applications:
See Walter, Skylab SL-3

A doped InSb crystal was processed to investigate the effects of the reduced gravity environment on the homogeneity of the dopant throughout the crystal. The distribution of the dopant has a major influence on the semiconductor characteristics. A crystal exhibiting a uniform distribution will have consistent electrical properties. On Earth, gravitational effects (i.e., convection) inhibit the growth of large crystals that have a uniform dopant distribution throughout the sample.

References/Applicable Publications:
(1) Walter, H. U.: Seeded, Containerless Solidification of Indium Antimonide (Skylab Experiment M-560). In Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. I, April 30-May 1, 1974, NASA Marshall Space Flight Center, Alabama, pp. 257-273. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 22-24, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) Naumann, R. J. Herring, H. W.: Experiment M560, Seeded, Containerless Solidification of Indium Antimonide. In Materials Processing in Space: Early Experiments, NASA SP-443, pp. 56-57. (post-flight)

(4) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(5) Experiment M560-Growth of Spherical Crystals. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-65 - 5-69. (post-flight)

(6) Walter, H. U.: Space Processing of Indium Antimonide Single Crystals by Seeded, Containerless Solidification: Skylab Experiment M-560 Final Report. July 1978, University of Alabama in Huntsville (UAH) Research Report No. 190, 74 pp. (post-flight)

(7) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(8) Growth of Spherical Crystals (M560). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-50. (post-flight; very short summary)

(9) Bourgeois, S. V. and Spradley, L. W.: Thermocapillary Convection in Microgravity Crystal Growth Melts of Indium-Antimonide. Letters in Heat and Mass Transfer, Vol. 3, 1976, pp. 193-304. (related research)

(10) Walter, H. U.: Containerless Processing of Single Crystals. AIAA/AGU Conference on Scientific Experiments of Skylab, Huntsville, Alabama, October 30-November 1, 1974, AIAA Paper No. #74-1241, 9 pp. (post-flight)

(11) Naumann, R. J. and Mason, D.: Seeded, Containerless Solidification of Indium Antimonide. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 28-29. (post-flight)

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Experiment Origin: USA
Mission: SPAR 3
Launch Date/Expt Date: December 1976
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Electromagnetic levitation furnace located in the NASA Electromagnetic Containerless Processing Payload (ECPP) module
Builder of Processing Facility: Unknown

Experiment:
Containerless Processing of Beryllium (74-48)

Cast beryllium is a coarse-grained, brittle material. Before the material can be used for most applications, a hot working process is required. On Earth, grain refiners (e.g., beryllia, BeO) are mixed with the beryllium to try to produce a fine-grained (less than 100 microns) ductile material suitable for high-temperature applications. However, "Beryllia particles in molten beryllium agglomerate and segregate from the melt.... This is primarily due to Stokes collisions and velocity gradient collisions. As the Stokes velocity is directly proportional to 'g,' the acceleration due to gravity, the rate of agglomeration and subsequent segregation of beryllia from the melt ought to be considerably reduced in the weightless environment of space. As some of the fluid motion leading to velocity gradient collisions may arise from gravity-driven natural convection, the rate of agglomeration and segregation of beryllia from the melt ought to be further reduced in the weightless environment of space." (3, p. 59)

This SPAR 3 experiment was designed to study the solidification of a Be-BeO alloy under low-gravity conditions. The specific objective of the experiment was to prepare a cast beryllium alloy (with enhanced service properties) using BeO as a grain refining agent.

The experiment was performed an electromagnetic levitation furnace housed within the Electromagnetic Containerless Processing Payload (ECPP). During the mission, a 0.922 cm diameter, Be-1.5

wt.% BeO sample was suspended in the electromagnetic field of a cusp coil. The cusp coil, which consisted of adjacent coils which had opposing, alternating magnetic fields, was used to melt the sample by induction heating. Thermal control was set by timers such that the experiment sequence was (1) application of full power, (2) reduction of power, and (3) low power mode. In the low power mode, enough power was supplied to maintain the sample position against residual acceleration forces.

Reportedly, "Although one of the power amplifiers experienced a failure, this occurred after melting and although this reduced the superheat of the melt, the experiment proceeded as planned." (3, p. 59)

Post-flight examination of the flight sample revealed a uniform distribution of BeO particles. No portion of the sample was either heavily agglomerated or devoid of BeO. In contrast, the corresponding ground-processed samples showed a large amount of agglomeration and segregation. (Corresponding ground-based studies were performed using an apparatus similar to the flight processing facility which was constructed by The General Electric Company.)

Unexpectedly, the flight sample contained large grain sizes: averaging about 700 microns. It was concluded, therefore, that the addition of BeO did not produce finer grains or retard grain growth. The reasons for the lack of grain refinement may have been because the particles (1) were not stable in the melt, (2) did not possess a maximum surface area, or (3) did not have optimum surface characteristics. "It may be possible that as a result of the long molten dwell, the particles change their surface character as well as become inactive as sites for heterogeneous nucleation; however, this could not be observed at 30 000X. An optimum surface character... might be a rough or pitted surface." (1, p. VI-47)

It was reported that further research would be required to determine if BeO could be used as a grain refiner for beryllium.

Key Words: Containerless Processing, Containerless Melt, Electromagnetic Levitator, Magnetic Fields, Composites with Solid Particles, Binary Systems, Melt and Solidification, Superheating, Casting, Particle Dispersion, Particle Distribution, Particle Agglomeration, Collisions, Stokes Flow, Grain Structure, Grain Size, Nucleation, Segregation, Buoyancy-Driven Convection, Surface Morphology, Surface Roughness, Ductility, Brittleness, In-

duction Heating, Radiative Cooling, Acceleration Effects, High Temperature Materials, Solid/Liquid Dispersion, Solid/Liquid Interface, Liquid/Gas Interface, Hardware Malfunction

Number of Samples: one
Sample Materials: Be-1.5 wt.% BeO (KBI HIP-50 alloy) (Be*O*)
Container Materials: not applicable

Experiment/Material Applications:
When beryllium is processed with a grain refiner under reduced gravity conditions, a fine-grained, ductile material suitable for high-temperature applications may result.

References/Applicable Publications:
(1) Wouch, G., Keith, G. H., Frost, R. T., and Pinto, N. P.: Containerless Processing of Beryllium. In Space Processing Applications Rocket Program, SPAR III-Final Report, NASA TM-78137, pp. VI-1 VI-62, January 1978. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

(3) Wouch, G.: Containerless Processing of Beryllium. In Applications of Space Flight in Materials Science and Technology. Proceedings of a Conference Held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, issued September 1978, pp. 59-66. (appears to be this mission; post-flight)

(4) Containerless Processing of Beryllium. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, January 1979, NASA TM-78217, pp. 21-22. (post-flight)

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Experiment Origin: USA
Mission: SPAR 4
Launch Date/Expt. Date: June 1977
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Electromagnetic Containerless Processing Payload (ECPP)
Builder of Processing Facility: General Electric Company, Space Division, Space Sciences Laboratory, King of Prussia, Pennsylvania

Experiment:
Containerless Production of Bulk Metallic Glasses (74-49)

"When a metal or alloy solidifies, it usually divides into many small crystals. The atoms in each of these crystals are arranged in a periodic fashion known as a crystal lattice. Certain metal alloys, however, can be cooled so fast that the atoms do not have time to arrange themselves in a regular fashion but are instead arranged in a more or less random fashion like the atoms in ordinary glass. Such disordered materials are termed amorphous and have very different properties from the same material in a crystalline state. Present techniques for fast cooling of metals on Earth require that the metal be in very thin ribbon form so that heat can be extracted quickly." (3, p. 43)

Amorphous metallic alloys (metallic glasses) have, in the past, been prepared by (1) splat cooling, (2) roller quenching, and (3) quenching in water. Methods 1 and 2 induce quenching rates on the order of 10^4 to 10^6 ^oC/sec while method 3 usually results in a quench rate of 10^2 to 10^3 ^oC/sec. It was, therefore, proposed that the elimination of container walls, which can act as nucleation sites for crystalline growth, could allow production of metallic glasses with slower cooling rates (less than 10^2 ^oC/sec). In a reduced gravity environment (where such containerless processing is possible), "...the metal can be cooled below its usual melting point so that when freezing does finally take place, the liquid will be so viscous that the atoms in the liquid cannot rearrange themselves into a crystal." (3, p. 43) The ultimate result of this process would be an amorphous metal produced in a bulk form.

This SPAR 4 experiment was designed to produce a bulk specimen of ferromagnetic glass during low-gravity, containerless solidification. The specific objectives of the experiment were to (1) obtain a large degree of supercooling of a containerless melt of Fe-40 at.% Ni-14 at.% P-6 at.% B (Allied Chemical's METGLAS 2826), and (2) obtain the specimen by supercooling to the glass temperature, T_g, and thus avoid crystallization of a (possibly) highly supercooled containerless melt of this composition.

During the SPAR 4 sounding rocket mission, the Electromagnetic Containerless Processing Payload (ECPP) was to be used to melt and resolidify the Fe-Ni-P-B spherical sample. "The ECPP... was a facility utilizing eddy current forces induced by specially designed positioning coils to position and hold a molten metallic specimen. The specimen was also melted by the induced eddy currents, which resistively heated the specimen to melting temperature. Cooling would occur when the current was just reduced to the point where positioning was maintained." (Reference (4))

Reportedly, the experiment was not performed as planned. "Analysis of the flight experiment data indicated that the specimen was not melted during the flight. The input power to the specimen was too low... and the specimen load was not matched properly to the r.f. tank circuit. Metallographic analysis confirmed this failure to melt. The specimen had reached a temperature near melting and incipient melting had begun on the surface but had not progressed inward during the experiment." (1, p. IV-7)

Extensive ground-based solidification experiments were performed and the results are presented in detail in Reference (1). It was reported that "The ground based experimental work yielded new results in terms of understanding the visco-elastic properties of these glasses and the variation of viscosity through the glass transition temperature from metallic glass to crystalline solid. This last result is of importance in predicting what glasses can be produced at lower quench rates...." (1, p. IV-2)

Key Words: Containerless Processing, Electromagnetic Levitation, Magnetic Fields, Containerless Melt, Metallic Glasses, Ferromagnetic, Amorphous Materials, Metals and Alloys, Melt and Solidification, Crucible Effects, Spheres, Free Surface, Resistance Heating, Cooling Rate, Viscosity, Solid/Liquid Interface, Liquid/Gas Interface, Supercooling, Mechanical Strength, Hardness, Incomplete Sample Processing, Hardware Malfunction

Number of Samples: one
Sample Materials: Allied Chemical's METGLAS 2826, Fe-40 at.% Ni-14 at.% P-6 at.% B
(Fe*Ni*P*B*)
Container Materials: not applicable.

Experiment/Material Applications:
Ferromagnetic metallic glasses (amorphous ferromagnetic alloys) are magnetically "soft"; because of their low coercive force and high permeability, they can be easily magnetized. The glasses also possess high strength and high hardness properties.

"The choice of METGLAS 2826 composition was made for the following reasons: (1) examination of the many candidate compositions indicated that the prospects for success were good, because of the high reduced glass temperature (ratio of glass transition temperature to melting temperature); (2) the ferromagnetic metallic glass produced by roller quenching in the form of a 'tape' has been well characterized; (3) it has not yet been prepared in bulk form, either by water quenching or any other technique." (1, p. IV-2)

References/Applicable Publications:
(1) Lord, A. E., Jr., Wouch, G., and Frost, R. T.: Preparation of Amorphous Ferromagnetic Materials Through Containerless Solidification. In Space Processing Applications Rocket Project SPAR IV-Engineering Report (Final), NASA TM-78235, pp. IV-i - IV-24, January 1980. (post-flight report)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI Inc., Technical Report 1308, March 1978. (acceleration measurements for SPAR 1-4)

(3) Descriptions of Space Processing Applications Rocket (SPAR) Experiments, NASA TM-78217, January 1979, pp. 43-44. (post-flight)

(4) Input received from Investigators A. E. Lord, Jr. and G. Wouch, August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt. Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module: TEM 02-1 (resonant tube levitator with isothermal resistance-heated tube furnace)
Builder of Processing Facility: Levitator: Battelle-Institute, Frankfurt, Germany; furnace: unknown

Experiment:
Primary Crystallization of a Hypereutectic Antimony-Silver Alloy

On Earth, gravity-driven segregation and thermal convection induce the transport of primary crystals through the melt. This transport is thought to aid the growth of the crystals. In the low-gravity environment, such segregation, convection, and transport are reduced and diffusion-controlled growth of the primary crystals is expected.

This TEXUS 1 experiment was designed to (1) study how a hypereutectic alloy is influenced by diffusion-controlled growth and (2) determine if diffusion-grown primary crystals are smaller in size than corresponding Earth-grown crystals.

During the TEXUS 1 mission, an 8-mm diameter spherical sample of hypereutectic alloy Ag-60 wt.% Sb was melted and solidified under low-gravity conditions using the TEXUS Experiment Module TEM 02-1. The module consisted of an isothermal, resistance-heated tube furnace and resonant tube levitator. The levitator was designed to produce sound waves to suspend the sample during processing. A cine camera was used for sample observation.

Post-flight, it was reported that the "...acoustic levitator did not work as expected. The sample was processed while free-floating but it touched the prepositioning wire cage before it solidified." (3, p. 210)

Metallographic analysis of the sample indicated that no gravity-driven segregation occurred. The primary crystals were homogeneously distributed and had a very fine eutectic structure (the Sb-Ag structure was finer than that observed in 1-g processed materials). Surprisingly, the primary crystals were larger in the flight sample than in the ground-based reference sample. It was concluded that it is primarily the diffusion-

controlled growth that determines the size of the primary crystals. "The undisturbed solidification [smaller fluctuations of atoms near the surface of the growing crystal] achieved under microgravity conditions resulted in bigger primary crystals and deeper undercooling, therefore in a finer structure of the eutectic." (3, p. 210)

<Note: E.G. Lierke was the Principal Investigator for the operation of the acoustic positioner; H. Ahlborn was the Principal Investigator for the Ag-Sb sample analysis. For details concerning the performance of the TEM 02-1 Acoustic Positioner, see Lierke, TEXUS 1 (Chapter 18).>

<Note: Reference (2) was not available to aid in the preparation of this summary. Further, only the abstract of Reference (1) was used (the remainder of the document was written in German).>

<Note: Ahlborn has other experiments within this database related to solidification phenomena (see Löhberg, SPAR 2 (Chapter 17); Ahlborn, Spacelab 1 and D1 (Chapter 17)).>

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Hypereutectics, Eutectics, Metals and Alloys, Binary Systems, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Resistance Heating, Isothermal Processing, Undercooling, Spheres, Segregation, Buoyancy-Driven Convection, Thermal Convection, Diffusion-Controlled Growth, Diffusive Mass Transfer, Free Surface, Solid/Liquid Interface, Liquid/Gas Interface, Material Interaction with Containment Facility, Collisions, Sample Microstructure, Sample Homogeneity, Processing Difficulties, Hardware Malfunction

Number of Samples: one
Sample Materials: Ag-60 wt.% Sb; an inert gas mixture (krypton/helium) was also involved (see Lierke, TEXUS 1).
(Ag*Sb*, Kr*He*)
Container Materials: not applicable

Experiment/Material Applications:
The suppression of gravity-induced segregation and convection was sought during this low-gravity sounding rocket experiment. Such reduction was thought to result in smaller primary crystals within the melt.

References/Applicable Publications:

(1) Ahlborn, H.: Primary Crystallization of a Hypereutectic Antimony Silver Alloy Under Reduced Gravity. A Melting Experiment of the Texus I Project, TSLP Final Report, BMFT-FB-W-79-32, 1979. (in German; English abstract)

(2) Ahlborn, H.: Abschlussbericht TEXUS 1, Koln, DFVLR, 1978.

(3) Solidification of a Hypereutectic Alloy. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 210-211. (post-flight)

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Co-Investigator(s): None
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Experiment Origin: USA
Mission: SPAR 6
Launch Date/Expt. Date: October 1979
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: High temperature single axis levitation furnace (silicon carbide element furnace with a single-axis acoustic positioning apparatus)
Builder of Processing Facility: Intersonics, Inc., Northbrook, Illinois

Experiment:
Containerless Processing of Glass (74-42)

During the production of glasses on Earth, samples are usually contained within crucibles. During the melting of the samples, crucible constituents often react with sample constituents, chemically contaminating the glass. As the glass solidifies during the later part of processing, heterogeneous nucleation sites often result because the material is in contact with the container wall. Glasses which are processed in a low-gravity environment do not have to be contained. It is therefore expected that defects attributed to the container should be avoided.

This SPAR 6 experiment was the first in a series of investigations designed by Happe et al. to study the low-gravity containerless solidification of a glass. The principal objective of the experiment was to evaluate the capability of the experimental equipment to position, melt, and solidify a glass sample under flight conditions.

The experiment hardware consisted of (1) a furnace with silicon carbide elements, (2) a single-axis acoustic positioning device, (3) a Pt-30% Rh injection cage, and (4) a motion picture camera. A massive copper cooling shroud was used for radiation cooling of the sample. Other equipment associated with the hardware included (1) electronics, (2) timers, (3) temperature control equipment, (4) sample injection mechanism, and (5) furnace wall gates.

During approximately 4 minutes of the SPAR 6 low-gravity rocket phase, a single 1/4" diameter glass sphere of 39.3 Ga_2O_3-35.7 mol% CaO-25.0 mol% SiO_2 was (1) injected into the furnace hot zone, (2) completely melted and soaked at 1575 ^{o}C, and (3) cooled

via radiation (by placing the cooling shroud into the hot zone of the furnace).

Post-flight analysis of the camera film revealed that the sample "...touched the cage four times in the first 9 seconds,... remained in suspension for 27 seconds, at which time it drifted to the injection cage and attached itself to one of the platinum alloy wires making up the cage. It remained attached and centered on the cage wire during the remainder of the processing cycle." (1, p. III-1)

Post-flight analysis of the sample revealed a spherical shape except for protrusions, caused by the wetting of the wire by the melt. These protrusions were located at either pole where the Pt emerged from the material. <Note: It appears (from Reference (1)) that three small bubbles were also located in the sample near the wire.> Numerous small crystal rosettes, which could not be seen macroscopically, were located on the sample surface. Analysis of the rosettes indicated that they had nearly the same composition as the glass with the principal constituent being $Ca_2Ga_2SiO_7$. Platinum (with some Rh present) was reported as the probable cause of the crystal nucleation. The most likely cause of Pt presence was either "...(1) mechanical transfer from the loose-fitting injection cage during lift-off of the rocket or (2) surface diffusion from the platinum-rhodium wire which was in contact with the glass sample during most of the flight experiment cycle." (1, p. III-2) It was concluded that Pt contamination of the sample surface and subsequent crystallization would be reduced if (1) the Pt cage firmly gripped the sample (prior to experiment initiation) to eliminate its rattling during lift-off and (2) the acoustic positioning device prevented contact with the injection cage during processing.

Conclusions related to the performance of the experimental apparatus included:

(1) The furnace and heating elements survived the launch environment.

(2) The flight sample was completely melted during the flight indicating that the processing temperature time cycle was adequate.

(3) "The acoustic positioning device was able to capture the sample, despite a rough injection. After several contacts with the cage during the first nine seconds, the acoustic positioner satisfactorily prevented contact of the sample with furnace components for 27 seconds before it contacted the cage wire." (1, p. III-43)

(4) "The copper cooling shroud was successfully deployed and cooled the sample approximately to the shroud temperature in the allotted time." (1, p. III-43)

(5) The motion picture camera functioned "flawlessly."

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Glasses, High Temperature Materials, Model Materials, Ternary Systems, Spheres, Sphericity, Free Surface, Wetting, Material Interaction with Containment Facility, Collisions, Nucleation, Thermal Soak, Viscosity, Radiative Cooling, Solid/Liquid Interface, Liquid/Gas Interface, Bubbles, Contamination Source, Surface Morphology, Optics Applications, Acceleration Effects, Rocket Vibration

Number of Samples: one
Sample Materials: silica modified gallia-calcia glass 39.3 Ga_2O_3-35.7 CaO-25.0 SiO_2, mol%
(Ca*O*Ga*O*Si*O)
Container Materials: not applicable

Experiment/Material Applications:
A silica-modified gallia-calcia material was used for this experiment due to its ability to solidify as a glass. The binary gallia-calcia composition has a very low viscosity, making it difficult to solidify in other than a crystalline condition. The addition of silica results in a material which can solidify as a glass.

The experimental material was used as a model system for special-purpose optical glasses.

References/Applicable Publications:
(1) Happe, R. A.: SPAR VI Experiment Report-Containerless Processing of Glass-Experiment 74-42. In Space Processing Applications Rocket Project SPAR VI Final Report, NASA TM-82433, October 1981, pp. III-1 - III-54. (post-flight)

(2) Ray, C. S. and Day, D. E.: Description of the Containerless Melting of Glass in Low Gravity. The National Society for the Advancement of Material and Process Engineering, SAMPE Technical Conference, Cincinnati, Ohio, October 4-6, 1983, pp. 135-145.

(3) Happe, R. A.: Manufacturing Unique Glasses in Space-First Interim Report. Rockwell International Corporation, Space Division, SD 74-SA-0174 (November 1974). (preflight)

(4) Happe, R. A.: Manufacturing Unique Glasses in Space-Second Interim Report. Rockwell International Corporation, Space Division, SD 76-SA-0029 (March 1976). (preflight)

(5) Whymark, R., Rey, C., Yearnd, J., and Broz, R.: Acoustic Levitation Materials Processing Systems. 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 15-17, 1979, AIAA Paper #79-0370, 9 pp. (discussion of apparatus)

(6) Input received from D. E. Day, July 1988 and July 1993.

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Experiment Origin: USA
Mission: SPAR 8
Launch Date/Expt. Date: November 1980
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: High temperature single-axis levitation furnace (silicon carbide element furnace with a single-axis acoustic positioning device)
Builder of Processing Facility: Intersonics, Inc., Northbrook, Illinois

Experiment:
Glass Formation Experiment (74-42/1R)

This SPAR 8 experiment was the second in a series of investigations designed by Happe et al. to study the low-gravity containerless solidification of a glass (see Happe, SPAR 6). The overall objective of the experiment was to evaluate the capability of the employed experimental equipment to position, melt, and solidify a glass sample under flight conditions. Because of earlier hardware difficulties on SPAR 6, additional objectives of the SPAR 8 experiment included (1) increasing the sonic positioning time to encompass the entire heating and cooling cycle, and (2) eliminating sample platinum contamination.

The experiment hardware was similar to that used by Happe during the SPAR 6 experiment (see Happe, SPAR 6 for a detailed hardware description); only the acoustic positioning device and Pt injection cage wire had been changed for the SPAR 8 mission.

During the earlier SPAR 6 experiment by Happe, a single spherical sample of a silica-modified gallia-calcia glass was melted and solidified in a single-axis acoustic positioning device. Because the sample levitation time (during that mission) was much shorter than intended (27 seconds vs. the intended 240 seconds) and because this anomaly was suspected to have been due to a significant drop in sonic power, the acoustic positioning device was now equipped with a larger sonic driver. Further, because the SPAR 6 glass sample was contaminated with Pt (from the wires of the injection cage) and underwent an unexpected crystallization, the cage wires (in the area which gripped the sample) were covered with alumina beads. It was believed that the alumina would prevent the mechanical transfer of Pt to the sample.

During the SPAR 8 sounding rocket mission, the High Temperature Single-Axis Levitation Furnace was used to process a single 1/4" glass sample sphere of 39.3 Ga_2O_3-35.7 CaO-25.0 mol% SiO_2. The expected experiment timeline and processing temperatures were not detailed.

It was reported that the SPAR 8 rocket was successfully launched and achieved low-gravity conditions with normal flight experiment operation. However, during the re-entry phase, "The payload parachute opened prematurely at [a] higher-than-planned altitude resulting in knotting of the central portion of the parachute and tearing of the fabric. As a result of the parachute malfunction the payload impacted the desert floor at a velocity of approximately 300 ft./sec. (...[approximately] 200 MPH)." (1, p. IV-11) As a result of this malfunction, the experimental apparatus was completely destroyed and the glass sample could not be recovered. Therefore, elimination of Pt contamination of the sample could not be verified. However, the film recording of the experiment was located and processed.

Post-flight analysis of the film indicated that the sample impacted the sample cage during processing, resulting in its attachment to the cage wires. However, the sample remained levitated for 82 seconds, compared to the 27 seconds of the SPAR 6 experiment. It was reported that "Inadvertent water vapor contamination of the furnace chamber is believed by the equipment contractor to have weakened the levitation force and caused the sample to ultimately move out of the [sonic] pressure well." (1, p. IV-18)

<Note: The source of this water vapor was not discussed in the available publications. D. E. Day (see Reference (2) below) reported that the source of the water vapor was "...not exactly known. [The] Most likely source... [was] the porous insulation in the furnace wall. The moisture trapped in these pores appeared as water vapor during furnace heating.

"The intensity and peak position of the sonic field depends strongly on the nature of [the] atmosphere along with the temperature and pressure. The presence of water vapor might have caused the intensity to change and peak position to shift creating instability in the performance of the levitator." (2)>

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Glasses, Model Materials, Ternary Systems, High Temperature Materials, Spheres, Free Surface, Wetting, Coated Surfaces, Material Interaction with Containment Facility, Collisions, Thermal Soak, Radiative Cooling, Solid/Liquid Interface, Liquid/Gas Interface, Contamination Source, Vaporization, Payload Survivability, Payload Recovery System Failure, Optics Applications

Number of Samples: one
Sample Materials: Silica modified gallia-calcia glass 39.3 Ga_2O_3-35.7 CaO-25.0 SiO_2, mol%
(Ca*O*Ga*O*Si*O)
Container Materials: not applicable

Experiment/Material Applications:
See Happe, SPAR 6.

References/Applicable Publications:
(1) Happe, R. A. and Kim, K. S.: SPAR VIII Experiment Report-Containerless Processing of Glass-Experiment 74-42. In Space Processing Applications Rocket Project, SPAR VIII Final Report, NASA TM-82578, June 1984, pp. IV-i - IV-35. (post-flight)

(2) Input received from D. E. Day, July 1988 and July 1993.

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Co-Investigator(s): None
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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Experiment Assembly (MEA-A1) housed on a STS Payload Bay OSTA-2 Pallet Platform
Processing Facility: Single-Axis Acoustic Levitator (SAAL)/furnace equipped with automatic sample exchanger (the apparatus had been used in the SPAR Program).
Builder of Processing Facility: Intersonics, Inc., Northbrook, Illinois

Experiment:
Containerless Processing of Glass Forming Melts (81F01)

On Earth, the processing of glasses is often complicated by several factors including (1) buoyancy-driven convection and density-driven segregation in fluid melts, (2) heterogeneous nucleation/crystallization at the melt/container interface, and (3) chemical contamination from crucibles containing the highly corrosive glass melts. Containerless processing of glass forming melts in the reduced-gravity environment should minimize the convection and segregation, while eliminating sample material reactions with crucibles.

This containerless processing experiment was one of several materials processing investigations contained in the Materials Experiment Assembly (MEA-A1) on the STS-007 mission. (The MEA-A1 was located in the STS payload bay during the flight.) The experiment was the first in a series of investigations designed by Day et al. to study the heating of samples within a Single-Axis Acoustic Levitator/furnace (SAAL). Samples of interest included (1) spherical glass-forming materials and (2) nonmelting alumina.

The following objectives were to be accomplished during the section of the investigation which involved the heating of glass forming materials:

"(a) obtain quantitative evidence for the suppression of heterogeneous nucleation/crystallization in containerless melts,

"(b) study melt homogenization in the absence of gravity driven convection,

"(c) develop the procedures for preparing precursor samples that will yield bubble-free, high purity, chemically homogeneous melts in micro-g,

"(d) perform comparative property analysis of glasses melted on earth and in micro-g, and

"(e) assess the suitability of the levitator/furnace apparatus for processing multicomponent, glass forming melts in micro-g." (1, p. 4)

The following objectives were to be accomplished during the section of the investigation which involved the heating of nonmelting alumina:

(1) confirm positioning and position stability of samples within the levitator,

(2) determine the effects of acoustic streaming,

(3) allow comparison of the acoustic space experiments to 1-g experiments, and

(4) observe the levitator and sample-insertion-apparatus at maximum operating temperature (1550 ^{o}C).

A total of eight samples were employed in the experiments (see the **Sample Materials** section (below) for sample compositions). Three of these samples were nonmelting alumina and five were 6 mm diameter glass forming materials. During the mission, each sample was to be (1) inserted into the preheated furnace, (2) heated to the appropriate temperature (melting the glass samples), (3) cooled to the appropriate temperature via the insertion of a cooling shroud, and (4) removed from the furnace. (During the heating process, the operating temperature of the furnace was between 1000 ^{o}C and 1550 ^{o}C).

Reportedly, "Two samples were successfully levitated at high temperatures before the experiment was prematurely terminated. The principle of acoustically levitating fluid melts at elevated temperatures was demonstrated." (6)

Post-flight analysis of "...the data stored in the flight recorder showed that six of the eight samples were successfully inserted into and removed from the furnace. However, none of the sample[s] attained the desired temperature. No change in weight, color, physical appearance, or shape was found for any of the flight... samples, [when] compared to... [ground-prepared samples], except for... [sample 3, the glass forming] hot pressed

sample. This flight sample had partially melted and was stuck to the platinum cage wires. [The cage, which surrounded the samples during processing, caught the sample if it escaped from the acoustic well.] A large pore... [approximately] 3 mm in diameter was found inside the flight sample. This large pore is believed to have formed from the coalescence of many much smaller pores initially present in the hot pressed... [ground-prepared sample]. The motion picture camera failed to operate so no photographic evidence was obtained for the behavior of the samples inside the acoustic levitator/furnace.

"Because of the experiment malfunctions, none of the scientific objectives for experiment were attained in the MEA/A-1 experiment. Nevertheless, the knowledge gained from this experiment will be of value in the planning and execution of future flight experiments." (1, p. 2)

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Acoustic Streaming, Resonant Frequency, Glasses, Glass Formation, High Temperature Materials, Binary Systems, Ternary Systems, Spheres, Free Surface, Material Interaction with Containment Facility, Collisions, Buoyancy-Driven Convection, Segregation, Nucleation, Heterogeneous Nucleation, Radiative Cooling, Solid/Liquid Interface, Liquid/Gas Interface, Bubbles, Bubble Coalescence, Pores, Surface Morphology, Sample Purity, Sample Homogeneity, Incomplete Sample Processing, Photographic Difficulties

Number of Samples: eight
Sample Materials: Samples 1, 2, and 8: alumina (Al_2O_3). Samples 3 and 4: $39.3Ga_2O_3$-$35.7CaO$-$25SiO_2$ mol% composition. (Sample 3 was hot pressed and contained large SiO_2 particles. Sample 4 (devitrified glass) contained a colored droplet on the external surface.) Sample 5: 56 Ga_2O_3-44 CaO mol% glass (devitrified). Sample 6: 33.3 Na_2O-$66.7B_2O_3$ mol% glass containing gas bubbles. Sample 7: 45 Na_2O-55 SiO_2 mol% glass (devitrified).
(Al*O*, Ga*O*Ca*O*Si*O*, Ga*O*Ca*O*, Na*O*B*O*, Na*O*Si*O*)
Container Materials: not applicable

Experiment/Material Applications:
Containerless processing of glasses in the reduced gravity environment may result in the preparation of high temperature (2500-3000 ^{o}C) ultrapure materials which contain no chemical contamination from container materials.

If glass formation is enhanced (as envisaged) in reduced gravity through the suppression of heterogeneous nucleation/crystallization, entirely new glasses (possibly with interesting properties) can be prepared in low gravity, which are very difficult, if not impossible, to prepare on Earth.

References/Applicable Publications:
(1) Day, D. E. and Ray, C. S.: Final Report for MEA/A-1 Experiment 81F01 Conducted on STS-7 Flight June 1983, Containerless Processing of Glass Forming Melts. University of Missouri-Rolla, NASA-CR-171048, 48 pp. (post-flight report)

(2) Harris, E. G.: Materials Experiment Assembly (MEA) Acceleration Summary, STS-7. Marshall Space Flight Center, JA62-004, July 1984. (acceleration measurements)

(3) Kreidl, N. J., Day, D. E., and Ray, C. S.: Containerless Glass Processing in Space. Glastech. Ber., 56K, pp. 151-156, 1983. (preflight)

(4) Ray, C. S. and Day, D. E.: Description of the Containerless Melting of Glass in Low Gravity. National SAMPE Technical Conference Series, 15, pp. 135-145, 1983. (preflight)

(5) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), pp. 25-26. (post-flight)

(6) Input received from Principal Investigator D. E. Day, June 1988 and June 1993.

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Experiment Origin: USA
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Experiment Assembly (MEA-A2) located in the STS Payload Bay
Processing Facility: Single-Axis Acoustic Levitator (SAAL)/furnace
Builder of Processing Facility: Intersonics Inc., Northbrook, Illinois

Experiment:
Containerless Processing of Glass Forming Melts-Determination of Critical Cooling Rates and Melt Homogenization (ME SAAL)

This containerless processing experiment was one of several materials processing investigations contained in the Materials Experiment Assembly (MEA-A2) on the STS-030 mission. (The MEA-A2 was located in the STS payload bay during the flight.) The experiment was the second in a series of investigations designed by Day et al. to study the heating of samples within a Single-Axis Acoustic Levitator/furnace (see Day, STS-007). Samples of interest included (a) glass-forming materials and (b) nonmelting alumina.

The objectives of the D1 experiment were essentially the same as those outlined for Day's earlier STS-007 flight. The D1 experimental goals, however, also included determining if glass shells (suitable for laser fusion targets) could be processed in the low-gravity environment.

A total of eight spherical samples (each approximately 6 mm in diameter) were employed in the experiments (see the MATERIALS section for sample compositions). Two of these samples were nonmelting alumina and six were glass-forming materials. The alumina samples were selected so as to check the engineering performance of the SAAL in low-gravity. Each of the six glass-forming samples were selected to (1) achieve one or more of the objectives of the experiment as outlined in Day, STS-007, or (2) examine shell production feasibility. Reference (3) (p. 180) details the specific purpose of each sample.

During the D1 mission, the processing procedure was fully automated. Each sample was to be:

(1) released into the furnace, (2) positioned "...at the potential energy minimum of the sound field confined between sound source and reflector of the SAAL" (4, p. 242), (3) heated to the appropriate temperature, (4) cooled to the appropriate temperature, and (5) retrieved from the furnace.

Reportedly, only samples 1, 2, and 3 were processed as planned. Samples 4-8 were never inserted into the furnace and, thus, were not processed. It appears that a "...lack of liquid coolant for the flight hardware created an over temperature condition of the furnace which caused a premature shut-down of the furnace." (4, p. 242)

A high quality photographic record of the processing of the first three samples indicated that the release of the samples went as planned. After the release, the samples oscillated in the acoustic field for 40-50 seconds and then became almost stationary in the acoustic energy well. The samples remained stationary during and after melting but began to oscillate again during the cooling sequence.

The first sample, alumina, was used to verify SAAL performance (as discussed earlier). The second sample (calcia- gallia-silica) was found solidified on the wire cage which surrounded the sample within the SAAL during processing. (The cage was used to catch samples if, for some reason, they were not held within the acoustic well.) The photographic record indicated "...that the sample struck and adhered to the cage when the cooling gate of the SAAL opened...." (4, p. 243) Processing of the third sample (a shell: soda-lime silica glass containing a large air bubble) resulted in an ellipsoidal piece of solid glass rather than the expected highly spherical shell of uniform wall thickness.

It was concluded that:

1. "Two glass forming compositions calcia-gallia-silica and soda-lime-silica) were successfully melted and cooled to glass while levitated in space. The calcia-gallia-silica composition requires a cooling rate [of approximately]... 11 to 12 ^{o}C/s to form glass on earth for the sample size used in the space experiment. This cooling rate is 2 to 3 times higher than the cooling rate (...[of approximately] 4 ^{o}C/s) for the MEA/A-2 experiment which yielded a glass for this composition. Clearly, the glass formation tendency for this calcia-gallia-silica composition is increased by 2 to 3 times when melted in space without a container." (4, p. 249)

2. "A usable and homogeneous glass was obtained for the calcia-gallia-silica composition for the melting time and temperature used in the flight experiment. Initially this sample was made by hot-pressing crystalline powders and contained random heterogeneities of pure silica. Chemical homogenization of this low viscosity melt was reasonably fast even in the absence of gravity-driven convection. Hot pressing appears to be a feasible way to prepare chemically pure samples for materials in space." (4, p. 249)

3. "The comparison of selected properties for the space and earth-melted calcia-gallia-silica glasses showed that the refractive index, dispersion, and Abbe number are slightly different, but there is no detectable difference in their infrared transmission or crystallization behavior. For this particular glass composition, melting in space produced no important difference in properties." (4, p. 250) <Note: "An Abbe number is a parameter used to describe the quality of optical glasses. More specifically, it describes the way the refractive index of a glass changes with wavelength of light and increases as the change of refractive index with wavelength decreases." (10)>

4. "Since the bubble escaped from the soda-lime-glass shell when it was remelted in space, the feasibility of shaping a glass shell in microgravity has not been fully demonstrated. The reshaping of glass shells in space is still considered feasible once the reason responsible for the bubble motion is identified." (4, p. 250)

5. "The operation of the single axis acoustic levitator furnace in space for high temperature liquid samples was generally satisfactory. For the first time, liquid samples were successfully levitated in space at high temperatures (1250 to 1500 ^{o}C) for periods of 6 to 12 minutes." (4, p. 250)

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Acoustic Streaming, Resonant Frequency, Glasses, Glass Formation, Binary Systems, Ternary Systems, Spheres, Shells, Sphericity, Free Surface, Surface Tension, Material Interaction with Containment Facility, Collisions, Buoyancy-Driven Convection, Segregation, Particle Dispersion, Nucleation, Radiative Cooling, Solidification Rate, Solid/Liquid Interface, Liquid/Gas Interface, Bubbles, Bubble Motion, Fluid Oscillation, Surface Morphology, Sample Purity, Sample Homogeneity, Thermal Environment More Extreme than Predicted, Optics Applications, Hardware Malfunction

Number of Samples: eight
Sample Materials: Samples 1, 8: alumina (Al_2O_3); Samples 2 and 4: 39.3Ga_2O_3-35.7 CaO-25 SiO_2 mol% composition. (Sample 2 was hot pressed and contained large SiO_2 particles. Sample 4 was glass (devitrified) containing a colored droplet on the external surface.) Sample 3: hollow soda-lime-silica glass shell; Sample 5: 33.3 Na_2O-66.7 B_2O_3 mol% glass containing gas bubbles; Sample 6: 56 Ga_2O_3-44 CaO mol% glass (devitrified); Sample 7: 50 PbO-50 SiO_2 mol% hot pressed.
(Al*O*, Ga*O*Ca*O*Si*O*, Na*O*B*O*, Pyrex, Ga*O*Ca*O*, Pb*O*Si*O*)
Container Materials: not applicable

Experiment/Material Applications:
"Glass shells several millimeters in diameter have important applications in laser fusion technology, but are essentially impossible to fabricate on earth. As there is no buoyancy force in microgravity, a bubble initially present inside the glass should not necessarily escape when the glass is remelted in space. When a levitated glass shell is remelted without a container, surface tension forces should reshape the sample into a spherical shell perhaps of uniform wall thickness." (4, p. 241)

See also Day, STS-007.

References/Applicable Publications:
(1) Day, D. E. and Ray, C. S.: Final Report for MEA/A-2 Experiment 81F01 Conducted on STS-22 (61-A Spacelab D1) Flight, October 1985, Containerless Processing of Glass Forming Melts. University of Missouri-Rolla, NASA George C. Marshall Space Flight Center, Huntsville, Alabama, NASA TM-4069, Vol. 2, October 1988, pp. 537-556. (post-flight report)

(2) Ray, C. S. and Day, D. E.: Containerless Glass Forming Experiment in Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 35. (post-flight)

(3) Ray, C. S. and Day, D. E. : Glass Formation in Microgravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 179-187. (post-flight)

(4) Ray, C. S. and Day, D. E.: Glass Formation in Microgravity. In Materials Research Society Symposia Proceedings (eds. R. H. Doremus and P. C. Nordine), Vol. 87, 1987, pp. 239-251. (post-flight)

(5) Ray, C. S. and Day, D. E.: Glass Melting in Microgravity. In the Japan Society of Microgravity Application (JASMA), Vol. 7, No. 3, pp. 94-108, 1990. (post-flight)

(6) Day, D. E. and Ray, C. S.: Research on Containerless Melts in Space. In Opportunities for Academic Research in Low-Gravity Environment, Progress in American Institute of Astronautics and Aeronautics (eds. G. A. Hazelrigg and J. M. Reynolds), Vol. 108, Chapter 6A, 1986, pp. 165-192. (preflight)

(7) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements on D1)

(8) Materials Processing Experiments in Space: MEA-A2 Payload. In Brochure Available from Application Payload Projects, NASA/ Marshall Space Flight Center, Huntsville, Alabama.

(9) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 25-26. (post-flight)

(10) Input received from Principal Investigator D. E. Day, June 1988 and June 1993.

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Experiment Origin: USA
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck
Processing Facility: Acoustic Containerless Experiment System (ACES): resistance heated furnace with acoustic levitator
Builder of Processing Facility: Jet Propulsion Laboratory, Pasadena, California

Experiment:
Acoustic Containerless Experiment System (ACES)

During this STS-011 experiment, the containerless processing of a fluoride glass sample was investigated. The specific objectives of the research were to:

(1) examine the feasibility of using a three-axis acoustic levitator to position the sample in the space environment,

(2) process the sample and examine its characteristics while levitated,

(3) compare the melting and subsequent crystallization of the containerless (levitated) sample to the melting and subsequent crystallization of a contained (ground-based) sample,

(4) observe the molten space sample stability when vibrated,

(5) measure the molten space sample surface tension, and

(6) observe the characteristics (growth and shrinkage rates) of an air bubble in the glass.

The processing of the single sample took place in the Acoustic Containerless Experiment System (ACES). The ACES consisted of a three-axis acoustic levitator, a resistance heated furnace, and a camera for video documentation. The camera had a 2-cm diameter, cylindrical field of view (through the center of the

furnace/levitator).

During the experiment, a 1-cm-diameter zirconium-fluoride glass sample was levitated, heated from 80 ^{o}C to 600 ^{o}C (during 70 minutes), held at this temperature for 20 minutes, and cooled.

Post-flight examination of the video record indicated that the sample did not remain within the camera's field of view at all times. At one point of the experiment, the sample (1) could be seen oscillating considerably, (2) was then lost from view, and (3) later returned. Upon its return, "The sample surface showed changes... with some possible strings or bumps emanating from its surface." (3, p. 2)

Post-flight examination of the furnace chamber indicated that a single glass sample was not available for analysis; rather, pieces of the sample were found. It was surmised "...that after the last excursion from view the soft sample stuck to the furnace wall or more likely part of the [holding] cage. It then melted and crystallized on cooling; it broke up at some point and was shaken about in the chamber during the rest of the flight and the landing." (3, p. 4)

Sample pieces were examined by X-ray diffraction and scanning electron microscopy. Reportedly, the surface of some of the sample pieces revealed "...conchoidal fractures with no evidence for intergranular fractures. Apparently the grain size is quite small, probably much less than 1 μm in diameter." (3, p. 4)

The Principal Investigator reported that the space-based containerless processing had resulted in fine, uniform crystallization, while Earth-based contained processing had resulted in large crystals which grew into the melt from the container surface. It was further reported that this difference in crystalline structure was probably related to the differing 1-g and reduced-g patterns of heat flow nucleation at the glass surface.

Experimental observations which pertained to objectives (5) and (6) (as described above) were not presented in detail in the available references.

Key Words: Containerless Processing, Containerless Melt, Glasses, Heavy Metal Fluoride Glasses, Ternary Systems, Acoustic Positioning, Acoustic Levitation, Melt and Solidification, Resistance Heating, Drops, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscillation, Fluid Oscillation, Liquid Dynamic Response,

Bubbles, Bubble Shrinkage, Bubble Growth, Solid/Liquid Interface, Liquid/Gas Interface, Free Surface, Liquid Vibration, Heat Transfer, Surface Morphology, Sample Microstructure, Nucleation, Grain Size, Grain Structure, Crystalline Structure, Sample Purity, Material Interaction with Containment Facility, Collisions, Payload Survivability, Infrared Detector Applications, Fiber Optic Applications, Photographic Difficulties

Number of Samples: one
Sample Materials: Heavy metal fluoride glass, 62 mol% ZrF_4 - 33% BaF_2 - 5%LaF_3, (Zr*F*Ba*F*La*F*)
Container Materials: not applicable

Experiment/Material Applications:
Heavy metal fluoride glasses, such as the one selected for this experiment, are transparent to 8 μm in the infrared and have potential applications as optical fibers and infrared optical components.

On Earth, crystalline nucleation often occurs at container walls as the glass sample is cooled. In a reduced gravity environment, containerless processing of the sample should result in purer glasses which are less likely to crystallize upon solidification.

References/Applicable Publications:
(1) Bansal, N. P. and Doremus, R. H.: Surface Tension Of ZrF_4-BaF_2-LaF_3 Glass. Communication Of American Ceramic Society, Vol. 67, October 1984, p. C-197. (no space results)

(2) 41-B Tenth Space Shuttle Mission. NASA Press Kit, February 1984, p. 30. (very short description; preflight)

(3) Doremus, R. H., Mathew, J., and Bradner, T.: Fluoride Glass Experiments in the ACES Apparatus on STS-11. (document received from Principal Investigator.)

(4) Input received from Principal Investigator R. Doremus, June 1988 and July 1993.

(5) Doremus, R. H.: Containerless Processing of Fluoride Glass. In Space Commercialization: Platforms and Processing, edited by F. Shahrokhi, G. Hazelrigg, and R. Bayuzick, Vol. 127 of Progress in Astronautics and Aeronautics, AIAA, Washington, D.C., IBSN 0-930403-76-2. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 9
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02: (monoellipsoidal mirror furnace (ELLI) combined with a resonant tube acoustic levitator)
Builder of Processing Facility: Levitator: Battelle-Institute, Frankfurt, Germany; Furnace: MBB/ERNO, Bremen, Germany

Experiment:
Containerless Solid Solidification of a PdCuSi Sample

Prior to the solidification of an undercooled melt, nucleation processes must occur. Classical nucleation theory distinguishes between homogeneous and heterogeneous nucleation. During homogeneous nucleation, a nucleus can grow only if the energy associated with the free enthalpy difference between the solid and liquid exceeds the energy contribution of the newly formed interface. This energy level corresponds to the activation energy required for a stable or growing nuclei to exist.

During heterogeneous nucleation, other phases, either within the melt (e.g., impurities) or in contact with the melt surface (e.g., crucible wall), participate in the process. These phases significantly reduce the activation energy required for the formation of stable nuclei. Thus, before homogeneous nucleation is possible, heterogeneous nucleation decreases the amount of undercooling possible.

Since undercooled melts are thermodynamically in a non-equilibrium state (1) non-equilibrium solidification is possible and (2) formation of non-equilibrium phases results. These solid-state, metastable phases have physical properties which can significantly differ from equilibrium phases. Homogeneous nucleation depends on the thermodynamic properties of the melt and, therefore, is an intrinsic property. However, heterogeneous nucleation is extrinsic and, therefore, can be influenced by (controllable) experimental parameters. For example, containerless solidification (the elimination of container walls) can result in an increased undercooling of the melt.

This TEXUS 9 experiment was the first in a series of investigations designed by Herlach to study the containerless solidification of a glass-forming PdCuSi alloy. The specific objective of the experiment was to levitate, melt, and solidify the glass in a furnace equipped with an acoustic positioning device.

In preparation for the rocket flight, a resonant tube acoustic levitator was configured with the mirror furnace ELLI and placed in the TEXUS Experiment Module TEM 02-2. The levitator had been partially tested during parabolic aircraft flights (low-gravity periods of approximately 10 seconds) and was to be further tested during the TEXUS 9 sounding rocket mission (low-gravity period of approximately 6 minutes).

Reportedly, a Pd 77.5 - Cu 6 - Si 16.5 sample (8 mm in diameter, 2.84 grams) was prepositioned in a wire cage in the focus of the mirror furnace. <Note: It was not clear if this sample composition was in weight, volume, or atomic percentage.> During the mission, "The experiment was performed in the temperature range 760 ^{o}C ([sample] melting point) to 1000 ^{o}C with heating rates of 10 to 20 ^{o}C/s. The sample was monitored with a CCD camera for automatic and telecommand control." (3, p. 362) <Note: The following is not clear: (1) if the sample was preheated prior to the launch or (2) at what stage of the experiment the above temperature range was realized.>

Reportedly, "The positioning test was only marginally successful. The sample was effectively free floating while completely melted. However, it started to rotate and oscillate during the cooling phase and came into contact with the wire cage several times. Finally, it remained attached to the cage while still molten and solidified." (3, p. 362)

Post-flight examination of the sample microstructure confirmed that the material was completely molten during the low-gravity portion of the mission. The Si contained in the sample had been oxidized indicating that the atmosphere was not sufficiently pure. Solidification was initiated when the sample contacted the wire cage. A lamellar eutectic structure formed at the wire/sample contact point. The lamellae increased in size with increasing distance from the contact point. This result indicated that at the point where the sample contacted the wire cage, both heterogeneous nucleation and a high cooling rate had occurred. Differential scanning calorimetry studies indicated that the undercooling prior to solidification had been less than 190 K. <Note: The amount of undercooling desired was not reported in the available publications.>

No other discussion concerning the experimental procedure or results could be located at this time.

<Note: Reference (1) (as listed below) was not available to aid in the preparation of this summary.>

<Note: D. M. Herlach was the Principal Investigator of the sample analysis; E. G. Lierke was the Principal Investigator of the acoustic positioner/furnace operation. For details concerning the performance of the acoustic positioner and furnace, see Lierke, TEXUS 9 (Chapter 18).>

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Glasses, Amorphous Materials, Glass Formation, Ternary Systems, Alloys, Spheres, Drops, Drop Oscillation, Drop Rotation, Sample Rotation, Liquid Vibration, Fluid Oscillation, Liquid Dynamic Response, Undercooling, Nucleation, Homogeneous Nucleation, Heterogeneous Nucleation, Sedimentation, Interface Physics, Sample Purity, Oxidation, Lamellar Eutectics, Solid/Liquid Interface, Liquid/Gas Interface, Sample Microstructure, Material Interaction with Containment Facility, Collisions, Crucible Effects

Number of Samples: one
Sample Materials: Pd-16.5% Si-6% Cu <Note: It was not clear if this sample composition was in weight, volume, or atomic percent.>
(Pd*Cu*Si*)
Container Materials: not applicable

Experiment/Material Applications:
The specific reason why the PdCuSi material was selected for this experiment was not detailed in the available publications.

"Containerless processing under high purity conditions favours the extension of the undercooling range of metallic melts and can result in metastable crystalline or amorphous (glassy) phases with unique physical properties. Microgravity conditions facilitate the containerless processing of samples and prevent the sedimentation of denser components." (3, p. 362)

References/Applicable Publications:
(1) Herlach, D. M., Gillessen, F., and Willnecker, R.: TEXUS 9, Abschlussbericht 1984.

(2) Herlach, D. M., Willnecker, R., and Gillessen, F.: Tiegelfreies Unterkühlen einer Pd-Cu-Si-Legierung. In Abschlussbericht TEXUS 13-16, 1988, pp. 94-96. (in German)

(3) Acoustic Positioning. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 362-363. (post-flight)

(4) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. Symposium in Industrial Activity in Space, Stressa, Italy, May 2-4, 1984, Proceedings, Paris, Eurospace, 1984, pp. 116-126. (preflight)

(5) Lierke, E. G., Grossbach, R., Flögel, K., and Clancy, P.: Acoustic Positioning for Space Processing of Materials Science Samples in Mirror Furnaces. In Proceedings of 1983 Ultrasonics Symposium, October 31-November 3, 1983, pp. 1129-1139. (preflight; mentions TEXUS 9 proposal; includes theoretical discussion)

(6) Clancy, P. F., Lierke, E. G., Grossbach, R., and Heide, W. M.: Electrostatic and Acoustic Instrumentation for Material Science Processing in Space. In Acta Astronautica, Vol. 7, 1980, pp. 877-891. (preflight; discusses apparatus; no results)

(7) Containerless Solidification of a Pd-Cu-Si Sample. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 364-365. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02: monoellipsoidal mirror furnace (ELLI) combined with an acoustic positioning device. (TEM 02 was previously flown on TEXUS 9, but was now modified to improve the radial acoustic positioning force approximately 50% over TEXUS 9.)
Builder of Processing Facility: Furnace: MBB/ERNO, Bremen, Germany <Note: It is not clear if the original builder of the levitator (Battelle-Institute, Frankfurt, Germany) also provided the newly modified acoustic positioner for this flight.>

Experiment:
Containerless Solid Solidification of a PdCuSi Sample

This TEXUS 14a sounding rocket experiment was the second in a series of investigations designed by Herlach to study the containerless solidification of a glass-forming PdCuSi alloy (see Herlach, TEXUS 9).

<Note: Details of the TEXUS 14a experimental setup and expected inflight experimental timeline were not discussed in the available publications. It appears that during the low-gravity experiment, the spherical eutectic alloy was to be acoustically positioned and heated using the TEXUS Experiment Module TEM 02-2. TEM 02-2 contained a monoellipsoidal mirror furnace with an acoustic positioning device.>

Reportedly, due to an unexpected "wobbling motion" of the TEXUS 14a rocket, uncontrollable accelerations were produced on the vehicle and the desired low-gravity level of 10^{-4} g was not attained. The experiment was reflown on TEXUS 14b (see Herlach, TEXUS 14b).

Documentation further detailing the results of this TEXUS 14a experiment does not appear to be available.

<Note: D. M. Herlach was the Principal Investigator of the sample analysis. E. G. Lierke was the Principal Investigator of the acoustic positioner/furnace operation (see Lierke, TEXUS 14a (Chapter 18)).>

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Metals and Alloys, Glasses, Amorphous Materials, Glass Formation, Ternary Systems, Alloys, Spheres, Undercooling, Nucleation, Solid/Liquid Interface, Liquid/Gas Interface, Sample Microstructure, Rocket Motion, Acceleration Effects

Number of Samples: unknown, probably one
Sample Materials: PdCuSi
(Pd*Cu*Si*)
Container Materials: not applicable

Experiment/Material Applications:
See Herlach, TEXUS 9.

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 53-55. (in German; post-flight)

(2) Experimentmodul TEM 02-2. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, p. 89. (in German; experiment Module)

(3) Lierke, E.-G., Grossbach, R., and Flögel: Final Report, ESTEC/Contract No. 5153/82/NL/HP. (experiment module modifications)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 14b
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02: monoellipsoidal mirror furnace (ELLI) combined with an acoustic positioning device. (TEM 02 was previously flown on TEXUS 9, but was now modified to improve the radial acoustic positioning force approximately 50% over TEXUS 9.)
Builder of Processing Facility: Furnace: MBB/ERNO, Bremen, Germany <Note: It is not clear if the original builder of the levitator (Battelle-Institute, Frankfurt, Germany) also provided the modified acoustic positioner.>

Experiment:
Containerless Solid Solidification of a PdCuSi Sample

This TEXUS 14b experiment was the third in a series of investigations designed Herlach to examine the containerless solidification of a glass-forming PdCuSi alloy (see Herlach, TEXUS 9, TEXUS 14).

The TEXUS Experiment Module TEM 02-2 was used for the experiment. The module contained a monoellipsoid reflecting furnace heated by a halogen lamp. An acoustic positioning device equipped with the furnace was used to position the spherical Pd-Cu-Si sample. (The acoustic positioning device had undergone improvements since its use during the TEXUS 9 mission. See Reference (4), p. 366, for a description of the improvements or Lierke, TEXUS 14b (Chapter 18).)

The sample was positioned at the pressure junction point of a stationary ultrasound wave. (The ultrasound wave was generated in a quartz tube connected to a sound transmitter.) Once positioned, the sample was melted and resolidified.

Observation of the sample was made possible by two CCD cameras positioned at right angles to each other. The sample position and lamp power output could be adjusted by remote control during the mission.

Post-flight examination of the film revealed that the sample melt did not contact any surface for a 40-second period. After an undercooling of about 170 K the sample began to solidify. Solidification was started by "...developing oscillations of the melt with subsequent contact with the cage boundary." (1, p. 95, translation)

Metallographic examination of the sample revealed two crystallization points. From these points, an eutectic structure grew into the sample. A sample processed on Earth, in a quartz crucible, exhibited an undercooling of 70 K. It was, therefore, concluded that containerless processing of this material results in an extension of undercooling. Auger analysis of the flight sample also revealed surface reactions of the melt with residual impurities in the furnace atmosphere.

<Note: E. G. Lierke was the Principal Investigator of the acoustic positioner/furnace operation. D. M. Herlach was the Principal Investigator of the sample analysis. Further discussion of (1) acoustic positioning device/furnace in flight operation and (2) source of the sample impurity can be found under Lierke, TEXUS 14b (Chapter 18).>

Key Words: Containerless Processing, Containerless Melt, Melt and Solidification, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Glasses, Amorphous Materials, Glass Formation, Ternary Systems, Alloys, Spheres, Drops, Drop Oscillation, Fluid Oscillation, Liquid Dynamic Response, Undercooling, Nucleation, Sedimentation, Interface Physics, Eutectics, Solid/Liquid Interface, Liquid/Gas Interface, Sample Microstructure, Surface Morphology, Impurities, Contamination Source, Material Interaction with Containment Facility, Collisions, Halogen Lamps

Number of Samples: one
Sample Materials: spherical sample of PdCuSi
(Pd*Cu*Si*)
Container Materials: not applicable

Experiment/Material Applications:
See Herlach, TEXUS 9

References/Applicable Publications:

(1) Herlach, D. M., Willnecker, R., and Gillessen, F.: Tiegelfreies Unterkühlen einer Pd-Cu-Si Legierung. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 94-96. (in German; post-flight)

(2) Experimentmodul TEM 02-2. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 89. (in German; experiment module)

(3) Lierke, E.-G., Grossbach, R., and Flögel, K.: Final Report, ESTEC/Contract Number 5153/82/NL/HP. (modification of experiment module)

(4) Acoustic Positioning. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 366-367. (post-flight)

(5) Containerless Undercooling of a Pd-Cu-Si Alloy. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 368-369. (post-flight)

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Co-Investigator(s): None
Affiliation(s): (1) Materials Processing Center, Massachusetts Institute of Technology, Cambridge, Massachusetts

Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Columbia)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Materials Science Laboratory (MSL-2) located in the STS payload bay
Processing Facility: Modified SPAR Electromagnetic Levitator (EML) with sample changer
Builder of Processing Facility: General Electric Company, Valley Forge, Pennsylvania

Experiment:
Alloy Undercooling/Electromagnetic Levitator (EML)

The solidification of a material requires the removal of (1) the specific heat of the melt and (2) the latent heat of fusion. Removal of this heat can be accomplished in a manner such that the material will not solidify when its normal solidification temperature is reached (the material is undercooled). Typically, solidification of metals occurs near their liquidus temperature and is induced by nucleation. Nucleation is promoted by (1) inclusions or impurities in the melt, (2) melt contact with the container, and/or (3) vibration of the experiment system. Reduced gravity, containerless-levitation of highly purified, metallic spheres may result in the suppression of the nucleation process. If this occurs, the degree of undercooling should increase and unique sample microstructures may result.

This objective of this STS experiment was to (1) levitate, melt, undercool, and solidify metallic spheres under low-gravity conditions and (2) examine of the resultant microstructures of the solidified samples.

The materials to be processed under low-gravity conditions consisted of six samples of three different Ni-Sn alloys (two samples of each composition): (1) Ni-32.5 wt.% Sn (eutectic), (2) Ni-25 wt.% Sn (hypoeutectic), and (3) Ni-37.5 wt.% Sn (hypereutectic). The samples were prepared from 99.99% pure Ni wire and 99.999% pure Sn shot. After machining (8.5 mm diameter), the samples were coated with Ferro brand EG-0222 silicate glass (0.15 mm thick). The glass coating served to (1) protect the samples during the long period between ground-based preparation and space processing and (2) act as a flux during processing to dissolve any metal oxides that could become

heterogeneous nucleation sites. (These sites would result in a decrease in the degree of undercooling).

The experiment was performed in the Electromagnetic Levitator Furnace (EML). Prior to the shuttle mission, the EML was contained in an experiment apparatus container and mounted on the Materials Science Laboratory (MSL). The MSL was then configured in the shuttle cargo bay.

The EML was designed to levitate a metallic sample within a cusp coil. The cusp coil consisted of adjacent coils with opposing magnetic fields. During an experiment run, the electromagnetic field of the coil heated and melted a sample by induction. After the sample was melted, the EML power was reduced and the sample cooled radiatively in the argon-filled chamber. A sample exchange system configured with the EML held the samples to be processed during the mission. This system consisted of (1) a specimen exchanger, (2) sample retaining wires, and (3) a sample collection cage. Photographic and pyrometric systems were available to monitor the sample during processing.

It was intended that the samples undergo three successive heating/cooling cycles, each cycle spaced 3 hours apart. Each cycle consisted of (1) levitating and stabilizing the sample (2 minutes, power level of 14 watts), (2) melting the sample (2 minutes, 600 watts), and (3) cooling and solidifying the sample (6 minutes, 14 watts).

Reportedly, difficulties prevented processing of all but one of the samples (the Ni-32.5 wt.% Sn alloy). "The power transistors of the power amplifier are attached to a heat sink through which cooling water circulates. A sensor attached to this heat sink measures its temperature, which is recorded every second and transmitted on the ground link.... [T]his temperature was observed to rise rapidly and exceeded saturation about 120 sec after full power was applied. It was also observed that the EML power output during the 120 sec dropped from 89 to 80 percent of the planned power level.... [F]ollowing the heating period, the power level dropped to below that necessary for levitating the specimen. Three hours later, when the second cycle for the first specimen was to begin, the EML delivered no radio frequency (RF) power, and the unit was turned off. The EML was turned on once more on the following day, but again no RF power was generated." (3, p. 4)

Post-flight, an investigation of the apparatus failure revealed that (1) the coolant loop contained gross particulate contamination which originated during the EML manufacture, (2) the particulates were probably dislodged during launch induced vibration and carried along by the coolant under low-gravity conditions,

and (3) the particles collected in the orifice/metering area of a regulating valve, inhibiting coolant flow, and resulting in an overheating of the power transistors. (There was also a problem with noise interference with the pyrometric temperature readings. See Reference (3) for this discussion.)

Post-flight examination of the pyrometer data pertaining to the partially processed specimen indicated "...that undercooling did not exceed 40 K, a disappointingly small amount, but in view of the problems encountered, logical." (3, p. 6) Examination of the sample surface revealed large, curved well-developed dendrites with primary, secondary, and tertiary arms. Since the sample was eutectic, the presence of dendrites was unexpected. The curved nature of the dendrites was attributed to "...thermal stresses within the dendrite resulting from steep thermal gradients during recalescence [reheating of the undercooled material due to the release of the heat of fusion], or mechanical stresses resulting from solidification shrinkage and convection during dendrite growth. The curvature of the dendrites is determined by heat flow and/or fluid flow, not by the preferred lowest energy orientation...." (3, p. 7)

The sample was also cross-sectioned and it was found that nearly 90% of its microstructure (volume fraction) was composed of normal lamellar eutectic. This amount of eutectic corresponded to an initial undercooling of less than 30 K. Faceted crystals were also located dispersed throughout the sample. It appeared that the eutectic may have nucleated on some of these crystals. Energy dispersive X-ray analysis indicated that the crystals contained a measurable amount of iron. However, the surrounding matrix contained no iron. The source of the iron was the retaining wire, which was composed of steel. The wire had embedded the sample while still molten and dissolved in the sample. This resulted in nucleation of the specimen and caused a lower undercooling.

Key Words: Containerless Processing, Containerless Melt, Electromagnetic Levitation, Magnetic Fields, Melt and Solidification, Heat of Fusion, Metals and Alloys, Metallic Oxides, Eutectics, Hypoeutectics, Hypereutectics, Binary Systems, Spheres, Free Surface, Coated Surfaces, Surface Morphology, Thin Films, Flux, Material Interaction with Containment Facility, Collisions, Heterogeneous Nucleation, Nucleation, Induction Heating, Undercooling, Radiative Cooling, Convection, Thermal Gradient, Saturation, Solid/Liquid Interface, Liquid/Gas Interface, Contamination Source, Sample Microstructure, Lamellar Eutectics, Faceted Eutectics, Microcrystalline Dispersion, Dendrites, Dendritic Solidification, Dendritic Arm Spacing, Sample Shrinkage, Hardware

Malfunction, Thermal Environment More Extreme Than Predicted, Rocket Motion, Rocket Vibration

Number of Samples: Six, although only one was actually processed.
Sample Materials: Two samples of each composition: (1) Ni-32.5 wt.% Sn, (2) Ni-25 wt.% Sn, and (3) Ni-37.5 wt.% Sn. All samples were coated with Ferro brand EG-0222 silicate glass (0.15 mm thick)
(Ni*Sn*)
Container Materials: not applicable

Experiment/Material Applications:
The Ni-Sn experiment material was selected because (1) it represents a simple eutectic system, (2) it has a wide temperature range of co-existing solid/liquid region, and (3) its alloys have been used for ground-based undercooling studies.

Space processing is desirable primarily for reduction of convection effects on nucleation and microstructure formation. The reduction of such effects may (1) result in a higher undercooling of the sample, and (2) produce a sample microstructure which is unaffected by gravity-induced mass transport processes.

References/Applicable Publications:
(1) Frost, R. T., Flemings, M. C., Szekely, J., El-Kaddah, N., and Shiohara, Y.: Electromagnetic Containerless Undercooling Facility and Experiments for the Shuttle. Advances in Space Research, Vol. 4, No. 5, 1984, pp. 99-103. (preflight)

(2) Materials Processing Experiments in Space: MSL-2 Payload. Document developed by Teledyne Brown Engineering under the direction of the Application Payload Project Office, Marshall Space Flight Center, Huntsville, Alabama, pp. 1-4. (MSL-2, processing facility)

(3) Harf, E. F., Piccone, T. J., Wu, Y., Flemings, M. C., Shiohara, Y., Gardner, L. B., and Winsa, E. A.: The Alloy Undercooling Experiment in the Columbia STS-61 C Space Shuttle Mission. AIAA Paper #87-056, American Institute of Aeronautics and Astronautics (1987), also NASA TM-88909.

(4) Piccone, T. J., Harf, F. H., Wu, Y., Shiohara, Y., Flemings, M. C., and Winsa, E. A.: Solidification of Undercooled Ni-Sn Eutectic Alloy Under Microgravity Conditions Aboard the Space Shuttle. In Materials Processing in the Reduced Gravity of Space, Materials Research Society Symposium Proceedings Vol. 87, R. H. Doremus and P. C. Nordine (eds.), MRS (1987), pp. 47-56.

(5) Electromagnetic Levitator Furnace. In Microgravity Science and Applications Experiment Apparatus and Facilities, document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 22-23. (processing facility)

(6) Input Received from Experiment Investigator, July 1988.

(7) Input Received from Yanzhong Wu (Massachusetts Institute of Technology), August 1993.

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CHAPTER 7

CRITICAL POINT PHENOMENA

Principal Investigator(s): Straub, J. (1), Lange, R. (2)
Co-Investigator(s): None
Affiliation(s): (1,2) Technische Universität, Munich, Germany

Experiment Origin: Federal Republic of Germany
Mission: TEXUS 6
Launch Date/Expt Date: May 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-7
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Phase Separation/Transformation

This TEXUS 6 experiment was the first in a series of investigations designed by Straub et al. to study phase separation and transformation under low-gravity conditions.

No documents (published in English) describing the experimental objectives, setup, or results from this investigation could be located.

<Note: References (3)-(5), which were not available to aid in the preparation of this experiment summary, may contain additional information about this investigation.>

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Binary Systems

Number of Samples: one
Sample Materials: SF_6
(S*F*)
Container Materials: experiment cell: unknown

Experiment/Material Applications:
See Straub, TEXUS 8 "Phase Separation/Transformation"

References/Applicable Publications:

(1) Nitsche, K., Straub, J., and Lange, R.: Ergebnisse des TEXUS-8 Experiments "Phasenumwandlung." Forschungsbericht Luft und Raumfahrt, BMFT (1984). (TEXUS 8, in German)

(2) Input received from B. Vogel (Technische Universität, Munich), August 1993.

(3) Straub, J. and Lange, R.: Untersuchung kritischer Phänomene unter reduzierter Schwerkraft-Werkstofforschung und Verfahrenstechnik unter Weltraumbedingungen; BMFT-Status-Seminar "Spacelab-Nutzung," Tübingen, Deutschland, 1978, DGLR-Bericht 79/01, Köln: DGLR, pp. 185-192. (in German)

(4) Lange, R. and Straub, J.: Das Verhalten fluider Stoffe im krit. Gebiet unter reduzierter Schwerkraft: Ein Experiment zur Bestimmung der isochoren Wärmekapazität-Werkstofforschung u. Verfahrenst. im Weltraum; BMFT-Status Seminar 1982, pp. 195-202.

(5) Lange, R. and Straub, J.: Phasenübergänge am kritischen Punkt fluider Systeme unter reduzierter Schwerkraft-Ergebnisse zweier vorbereitender Experimente; Kritische Erscheinungen, Phasenbildung und Transportvorgänge unter verminderter Schwerkraft, Richter, J. (Ed.), 18. Kolloq. d. Bunsen-Gesell., Aachen, pp. 24-33. (in German)

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Co-Investigator(s): None
Affiliation(s): (1,2) Technische Universität, Munich, Germany

Experiment Origin: Federal Republic of Germany
Mission: TEXUS 8
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-7: One fluid cell was employed for this part of the experiment.
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Phase Separation/Transformation Fluid-Gas
Part 1: Single Phase SF_6 Cooled to Two-Phase Region

"At the critical point, many physical properties of fluids are strongly modified and measurements of their behaviour can be difficult or inaccurate under Earth gravity." (4, p. 148) In a low-gravity environment, sample sedimentation and convection (which occur following phase separation during ground-based studies) are reduced, and the separation process is driven by mass diffusion and interface instabilities.

This TEXUS 8 experiment was the second in a series of investigations designed by Straub et al. to study phase separation and transformation under low-gravity conditions (see Straub, TEXUS 6). (The experiment was also the first of two phase-separation/transformation investigations performed by Straub on the TEXUS 8 mission.) The specific objective of the experiment was to investigate the vapor-liquid phase transition of sulfur hexafluoride, SF_6, at the critical density. (The experiment was performed in preparation of a later STS Spacelab D1 experiment.)

Prior to the initiation of the TEXUS 8 experiment, a fluid cell containing single-phase, sulfur hexafluoride vapor, SF_6, was held at a temperature 400 mK above the critical temperature, Tc. During the low-gravity flight, the cell was cooled to the two-phase liquid/vapor region (Tc - 400 mK). While temperature measurements were made at the center of the fluid and at the cell wall, the phase transition was documented by a 16 mm movie (cine) camera.

Reportedly, "During cooling it was observed that the sample uniformly assumed a yellow to brownish color. Approaching Tc it became increasingly darker and at Tc it was completely opaque. No brightening could be observed over a period of 3 minutes after crossing Tc to Tc - 400 mK. We interpret this observation as

being the critical opalescence near Tc and the formation of a dispersion of droplets or bubbles representing the initial state of the two new phases below Tc." (3, p. 246)

Reportedly (Reference (3)), the center cell temperature was found to lag the wall temperature in a manner that was similar to ground-based reference experiments. Thus, it was concluded (Reference (3)) that the temperature equilibrium was not hampered under reduced gravity for the cooling rate employed (2.6 mK/s). Further, it was reported (Reference (4)) that "The analysis of the temperature/time profile obtained showed no significant delay of the thermal response in the centre of the fluid on... cooling the container wall through the critical temperature. This indicated that the thermal equilibrium in the critical region was much more enhanced than had been expected, as the thermal diffusivity tends to be zero at Tc. This surprising result was particularly evident on the cine pictures where the homogeneous onset of the phase transition throughout the whole volume of the cell was observed." (4, p. 148) <Note: It is not clear if the last sentence of this quote applies to this experiment or to the other TEXUS 8 phase separation/transition experiment by Straub (see Straub, TEXUS 8, part 2).>

No further information concerning this experiment could be located at this time. <Note: Reference (1) (below) could not be attained prior to the preparation of this experiment summary. Further, it appears that the reference was written in German.>

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Binary Systems, Vapor/Liquid Transition, Sedimentation, Buoyancy-Driven Convection, Diffusive Mass Transfer, Thermal Diffusion, Thermal Equilibrium, Critical Density, Interface Stability, Interface Physics, Bubble Dispersion, Droplet Dispersion, Spinodal Decomposition, Nucleation, Cooling Rate

Number of Samples: one
Sample Materials: sulfur hexafluoride, SF_6
(S*F*)
Container Materials: experiment cell: unknown

Experiment/Material Applications:
A sample undergoing phase transition in a low-gravity environment should experience a reduction in sedimentation and convection. A reduction of these fluid transport contributions allows the investigation of spinodal decomposition and nucleation during the phase change.

This critical phenomena investigation is applicable to the study of chemical or electrical power technology and lubrication or solidification sciences.

References/Applicable Publications:
(1) Nitsche, K., Straub, J., and Lange, R.: Ergebnisse des TEXUS-8 Experiments "Phasenumwandlung." Forschungsbericht Luft und Raumfahrt, BMFT (1984).

(2) Nitsche, K. and Straub, J.: The Critical "Hump" of Cv Under Microgravity-Results from the D1 Spacelab Experiment "Wärmekapazität." In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 109-116. (TEXUS 8 experiments briefly mentioned on pages 113-114; post-flight)

(3) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near Critical Phenomena. In Fluid Sciences and Materials in Space, Edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 222-254. (TEXUS 8 experiments are specifically described on pages 245-246; post-flight)

(4) Phase Transition. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 148-149. (post-flight)

(5) Input received from B. Vogel (Technische Universität, Munich), August 1993.

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Co-Investigator(s): None
Affiliation(s): (1,2) Technische Universität, Munich, Germany

Experiment Origin: Federal Republic of Germany
Mission: TEXUS 8
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-7: One fluid cell was employed for this part of the experiment.
Builder of Processing Facility: ERNO, Bremen, Germany

Experiment:
Phase Separation/Transformation Gas-Fluid
Part 2: Two Phase SF_6 Heated to Single Phase Region

"At the critical point, many physical properties of fluids are strongly modified and measurements of their behaviour can be difficult or inaccurate under Earth gravity." (4, p. 148) In a low-gravity environment, sample sedimentation and convection (which occur following phase separation during ground-based studies) are reduced, and the separation process is driven by mass diffusion and interface instabilities.

This TEXUS 8 experiment was the second in a series of investigations designed by Straub et al. to study phase separation and transformation under low-gravity conditions (see Straub, TEXUS 6). (The experiment was also the second of two phase-separation/transformation investigations performed by Straub on the TEXUS 8 mission.) The specific objective of the experiment was to investigate the liquid-vapor phase transition of sulfur hexafluoride, SF_6, at the critical density. (The experiment was performed in preparation of a later STS Spacelab D1 experiment.)

Prior to the initiation of the TEXUS 8 experiment, a fluid cell containing two-phase, vapor/liquid sulfur hexafluoride, SF_6, was held at a temperature 400 mK below the critical temperature, Tc. During the low-gravity flight, the cell was heated to the single phase vapor region (Tc + 400 mK). While temperature measurements were made at the center of the fluid and at the cell wall, the phase transition was documented by a 16 mm movie (cine) camera.

Rotation of the fluid, attributed to the spin stabilization of the rocket was noted during the initial stages of the experiment. "...damping of this flow took approximately 1 minute. The two phases could easily be discerned, due to the difference of their refractive indices, as large inhomogeneous particles." (3, p. 246) It was also reported that "During heating, the critical

opalescence appeared at the boundary of the inhomogeneities as a dark colour.... The homogeneities could be observed up to Tc + 400 mK, but it can be observed that at the boundary a dissolving of the density inhomogeneities took place. The conclusion of this observation is that mixing in microgravity is a process which can take time." (3, p. 246).

Reportedly (Reference (3)), the center temperature was found to lag the wall temperature in a manner that was similar to ground-based reference experiments. Thus, it was concluded (Reference (3)) that the temperature equilibrium was not hampered under reduced gravity for the heating rate employed (2.6 mK/s). Further, it was reported (Reference (4)) that "The analysis of the temperature/time profile obtained showed no significant delay of the thermal response in the centre of the fluid on heating... the container wall through the critical temperature. This indicated that the thermal equilibrium in the critical region was much more enhanced than had been expected, as the thermal diffusivity tends to be zero at Tc. This surprising result was particularly evident on the cine pictures where the homogeneous onset of the phase transition throughout the whole volume of the cell was observed." (4, p. 148) <Note: It is not clear if the last sentence of this quote applies to this experiment or to the other TEXUS 8 phase separation/transition experiment by Straub (see Straub, TEXUS 8, part 1).>

No further information concerning this experiment could be located at this time. <Note: Reference (1) (below) could not be attained prior to the preparation of this experiment summary. Further, it appears that the reference was written in German.>

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Binary Systems, Vapor/Liquid Transition, Liquid/Vapor Interface, Particle Dispersion, Sedimentation, Buoyancy-Driven Convection, Diffusive Mass Transfer, Thermal Diffusion, Thermal Equilibrium, Critical Density, Interface Stability, Interface Physics, Rotating Fluids, Acceleration Effects, Rocket Motion, Fluid Motion Damping, Liquid Dynamic Response, Spinodal Decomposition, Nucleation

Number of Samples: one
Sample Materials: sulfur hexafluoride, SF_6 (S*F*)
Container Materials: fluid cell: unknown

Experiment/Material Applications:
A sample undergoing phase transition in a low-gravity environment should experience a reduction in sedimentation and convection. A reduction of these fluid transport contributions allows the investigation of spinodal decomposition and nucleation during the phase change.

This critical phenomena investigation is applicable to the study of chemical or electrical power technology and lubrication or solidification sciences.

References/Applicable Publications:
(1) Nitsche, K., Straub, J., and Lange, R.: Ergebnisse des TEXUS-8 Experiments "Phasenumwandlung." Forschungsbericht Luft und Raumfahrt, BMFT (1984).

(2) Nitsche, K. and Straub, J.: The Critical "Hump" of Cv Under Microgravity-Results from the D1 Spacelab Experiment "Wärmekapazität." In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 109-116. (TEXUS 8 experiments briefly mentioned on pages 113-114; post-flight)

(3) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near Critical Phenomena. In Fluid Sciences and Materials in Space, Edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 222-254. (TEXUS 8 experiments are specifically described on pages 245-246; post-flight)

(4) Phase Transition. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 148-149. (post-flight)

(5) Input received from B. Vogel (Technische Universität, Munich), August 1993.

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, M E D E A Double Rack
Processing Facility: High Precision Thermostat (HPT) containing a sample cell and four-stage scanning ratio calorimeter
Builder of Processing Facility: Kayser-Threde, Munich, Germany

Experiment:
Heat Capacity Near the Critical Point (MD-HPT-00)

"Studies of critical phenomena have grown to a considerable field in theoretical physics. In the recent years a global theory, the "renormalization group" has focused great expectations of scientists, for it predicts macroscopic thermodynamic properties around critical points without considering the molecular structure of the system to be investigated. This theory is applicable to physically different kinds of critical phenomena such as magnetisation [sic] of a solid body near the Curie Point as well as phase separations of binary mixtures or phase changes of pure liquids near critical temperature. The universality of this model has gained importance since up to the present day it has not been possible in most cases to describe the macroscopic behaviour including phase transitions out of molecular interaction using methods of statistical physics. However, the range in which the renormalization group holds is confined to a small region around critical points. This region is characterized such that macroscopic properties can be expressed in terms of simple power laws. For example the specific heat at constant volume c_v becomes...

$$[c_v/R = A((T-T_c)/T_c)^{-a} + B \qquad \text{eqn. (1)}]$$

where R denotes the gas constant, T_c the critical temperature and A,... [a], and B parameters for which theoretical calculation yield numerical values.

"Terrestrial investigations have been performed that show a reasonably good agreement of the experimentally determined parameters in Eqn. (1) with the corresponding theoretical results. Unfortunately, like many other measurements at fluid

critical states, these results suffer from gravitational influences that enhance their dominating significance just at the critical point, the target of all experimental efforts....

"The experimental determination of the parameters A,... [a], and B in the equation (1) for the isochoric specific heat c_v is inhibited by the fact that the power laws describe the behaviour of state only within a small temperature range $/t/ < 2*10^{-3}$ around the critical point where $t = (T-T_c/T_c)$.... Therefore, experimentation near critical state requires extraordinary efforts in thermostabilization, electronic measuring and control and can be performed only in samples of small dimensions. Fluid at critical temperature is very sensitive to external fields like temperature gradients and gravity forces." (4, p. 335)

This Spacelab D1 experiment was the third in a series of investigations designed by Straub et al. to study phase transition under low-gravity conditions (see Straub, TEXUS 6, TEXUS 8). The specific objective of the experiment was to measure the isochoric specific heat (c_v) of sulfur hexafluoride (SF_6) at its liquid-vapor critical point.

The experiment was performed in the D1 High Precision Thermostat (HPT). The HPT contained (1) a 30-mm diameter, 1-mm thick coin-shaped sample cell filled with SF_6 and (2) a four-stage scanning ratio calorimeter. During the mission, the calorimeter was used to determine the energy necessary to heat up 0.5 g SF_6 of critical density (0.737 g/cm^3) through the gas liquid critical point (from $T-T_c$ = -100 mK to $T-T_c$ = +100 mK).

Reportedly, "Despite the reduced experiment time of four days, four different heating rates (dT/dt = 3.6, 10, 20 and 100 mK/h) could be accomplished. Data cover temperature spans around the critical point of $/T-T_c/$ = 50 and $/t-t_c/$= 100 mK and were recorded with a density of 1 frame per 0.7 second." (8, p. 188)

Low-gravity specific heat (C_v) measurements were compared to theoretical predictions and ground-based measurements. While theory predicted that an enhanced peak in C_v (at the critical temperature) would occur in the low-gravity environment, the C_v vs. temperature curves were surprisingly below that predicted. The expected peak C_v degenerated to a slight hump. "In the four heating ramps the cell capacity C_0 [which is proportional to c_v] was measured and showed reproducibly a smooth shape in the critical region instead of a sharp peak." (8, p. 195) The low-gravity "peaks" were also lower than 1-g reference measurements. "These quite unexpected results cannot be explained by malfunction of hardware; after landing, the experiments were repeated in the original flight configuration and the critical peak was again observed. Wrong filling of the specimen cell or impurities of the

substance cannot explain this result.... The thermal equilibrium seems not to be greatly retarded... and therefore the mechanism of the mass transport from the liquid phase during heating and a possibly [sic] density inhomogeneity could be the only effects for the explanation at this moment. The finding is not well understood and still under investigation." (10, p. 244)

"The question whether relaxational effects are responsible for this unexpected behaviour cannot be answered so far. Therefore, we suggest for future experiments at the critical point to additionally measure temperature at several locations directly in the fluid sample to record an established or distorted thermal equilibrium. Furthermore, optical techniques are necessary to learn about the density distribution of fluid at critical state under reduced weight." (8, p. 195)

Further discussions of the governing theory, low-gravity experimental justification, experimental apparatus, data evaluation and results are detailed in several of the publications listed below.

<Note: Several references listed below were not available at the time this experiment summary was prepared.>

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Binary Systems, Vapor/Liquid Transition, Sedimentation, Buoyancy-Driven Convection, Diffusive Mass Transfer, Thermal Diffusion, Critical Density, Interface Stability, Interface Physics, Isochoric Specific Heat, Calorimetric Measurements, Scanning Ratio Calorimeter, Thermal Equilibrium, Heat Capacity

Number of Samples: one sample cell
Sample Materials: sulphur hexafluoride, SF_6
(S*F*)
Container Materials: stainless steel

Experiment/Material Applications:
"For theoretical modelling of critical phenomena the isochoric specific heat is one of the most important properties. Measurements of this quantity are only possible in the bulk fluid and due to the high compressibility[,] the results are strongly in-

fluenced by gravity." (10, p. 243)

"Studies of critical point phenomenon may have technological relevance to the chemical or electric power industries as well as to lubrication science, etc." (10, p. 223) See also experiment summary (above).

"SF_6 has been chosen [as the test fluid] because...

"- It is a test fluid of international interest....

"- Its molecule has spherical... [symmetry] thus making the comparison to theoretical calculations easier.

"- It is inflammable and nontoxic (Spacecraft Crew Safety).

"- Its critical parameters can be handled rather easily... thus lowering the efforts for pressure and temperature control." (9, p. 2)"

References/Applicable Publications:

(1) Nitsche, K. and Straub, J.: The Isochoric Specific Heat of Sulfur Hexafluoride SF_6 at the Critical Point Under μ-g Conditions. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 29. (abstract only; post-flight)

(2) Nitsche, K. and Straub, J.: Die isochore Wärmekapazität am kritischen Punkt unter reduzierter Schwere. In natur Wissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 370-373. (article in German; English abstract; post-flight)

(3) Straub, J., Nitsche, K., and Lange, R.: Specific Heat. In Scientific Goals of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 84-85. (post-flight)

(4) Nitsche, K., Straub, J., and Lange, R.: Isochoric Heat of Sulfur Hexafluoride at the Critical Point-A Spacelab Experiment for the German D1-Mission in 1985. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 335-340. (preflight)

(5) Nitsche, K. and Straub, J.: The Critical Hump of Cv Under Microgravity Results From the D1-Spacelab Experiment "Wàrmekapazitàt." In 6th European Symposium on Material Sciences Under Microgravity, Bordeaux, France, December 2-5, 1986, pp. 109-116. (post-flight)

(6) Lange, R. and Straub, J.: Die isochore Wàrmekapazitàt fluider Stoffe im Kritischen Gebiet-Voruntersuchungen zu einem Spacelab Experiment. BMFT-FB-W 84-034 (1984), 136 S. (in German; preflight; appears to be referring to this experiment)

(7) Straub, J., Lange, R., Nitsche, K., and Kemmerle, K.: Isochoric Heat of Sulfur Hexafluoride at the Critical Point: Laboratory Results and Outline of a Spacelab Experiment for the D1 Mission in 1985. International Journal of Thermophysics, Vol. 7, No. 2, 1986, pp. 343-356. (preflight)

(8) Nitsche, K. and Straub, J.: The Isochoric Specific Heat of Sulphur Hexafluoride SF_6 at the Critical Points Under μ-g Conditions. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 188-195. (post-flight)

(9) Kemmerle, K.: High Precision Thermostat: A set of Experiment Facilities for Caloric Research in Space. 27th Aerospace Sciences Meeting, January 9-12, 1989, Reno, Nevada, AIAA #89-041, 12 pp. (apparatus)

(10) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near Critical Phenomenon. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 221-256, specifically, pp. 243-244. (post-flight)

(11) Kemmerle, K.: Basic Design of the High Precision Thermostat (HPT) of the D1 Mission. In Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987. (apparatus)

(12) Straub, J. and Nitsche, K.: Isochoric Heat Capacity C_v at the Critical Point of SF_6 under Micro and Earth Gravity-Results of the German Spacelab Mission D1. 11th Symposium of Thermophysical Properties, Boulder, Colorado, June 1991, 24 pp. (post-flight)

(13) Grubic, M. and Kemmerle, K.: A Precision Lock-in Amplifier for Temperature Control in a Spacelab Calorimetric Experiment. J. Phys, E: Sci. Instrum., Vol. 18, 1985, pp. 572-574.

(14) Input received from B. Vogel (Technische Universität, Munich), August 1993.

(15) Die isochore Wärmekapazität am kritischen Punkt unter reduzierter Schwere - ein Experiment zur Ersten Deutschen Spacelab-Mission D1 1985; Jahrbuch der Technischen Universität München, pp. 118-127. (in German)

(16) Nitsche, K. und Straub, J.: Die isochore Wärmekapazität im kritischen Gebeit von SF_6 unter Erdschwere und reduzierter Schwere; BMFT Abschlussbericht, 1990.

(17) Kemmerle, K., Bernhardt, H.-G., Straub, J., Haupt, A., and Nitsche, K.: The High Precision Thermostat HPT-HYDRA. Proc. 8th European Symposium on Materials and Fluid Sciences in Microgravity, Brussels, Belgium, April 12-16, 1992, ESA-SP 333, pp. 361-366.

(18) Straub, J., Haupt, A., Nitsche, K., and Kemmerle, K.: A New Method for Hysteresis Measurements Developed for C_v Measurements at the Critical Point During the D-2 Mission. Proc. 8th European Symposium on Materials and Fluid Sciences in Microgravity, Brussels, Belgium, April 12-16, 1992, ESA-SP 333, pp. 301-308.

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Co-Investigator(s): None
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Experiment Origin: Great Britain
Mission: TEXUS 10
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-9: conductance autoclave
Builder of Processing Facility: The main facility for measurement and control: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany; autoclave: C.E.R.L., Leatherhead, Great Britain

Experiment:
Ionic Solutions Near the Critical Point of Water

Dissolved ions compress the solvent molecules in their vicinity - they electrostrict them. When a solvent has a high compressibility, as it will near its critical point (between 300 ^{o}C and the critical point), the effect is very significant and aqueous ionic solutions behave abnormally. One consequence is that solutions suffer gravitational sedimentation. This impedes study of the more fundamental critical point characteristics. "At present this behaviour is understood at best semi-qualitatively, so that solutions are very poorly characterized thermodynamically in this region. The result is that it is often impossible to make reliable statements concerning the role of the aqueous phase in certain corrosion and related phenomena. Such phenomena have potential importance in, for example, power station boilers." (7, p. 150)

This TEXUS 10 experiment was the first in a series of experiments designed by Turner to study ionic solutions near the critical point under low-gravity conditions. The specific objectives of the experiment were to (1) demonstrate that there is a gravitational effect on the solutions at the critical point, (2) assess the rate of desedimentation of the solution which occurs in the reduced gravity environment, (3) test the experimental equipment.

Before the mission, a 1.0 mM sodium chloride solution, contained within a 1-cm long, 0.5-cm inner diameter thick-walled sapphire tube, was configured within a conductance autoclave. Endcaps of the tube served as electrodes. <Note: The material composition of these endcaps as reported in the available references dif-

fered. For example, References (4) and (5) indicated that both electrodes were comprised of silver clad stainless steel; Reference (7) indicated that one of the electrodes was stainless steel and the other platinum.> Approximately 1.5 hours prior to the rocket launch, the temperature of the autoclave was ramped to 377 ^{o}C. It attained this temperature within 0.5 h and then remained at this value for 60 minutes prior to the launch.

During the mission, the the autoclave was heated by a precision furnace, and baffles usually ensured that the temperature difference from the top of the assembly to the bottom was less than 0.2 ^{o}C. Reportedly, the temperature was maintained at 377 +/- 1 ^{o}C.

During the low-gravity phase of the rocket flight, the conductance of the solution was measured. "The resistance of a 1 mm thick layer of solution between a 4 mm diameter PtRh electrode and one of the silver-clad stainless steel end caps of the cell was measured using 12 cycles of 30 s each. During each cycle, the measurements were made successively at 1, 0.1 and 10 kHz. The voltage was 40 mV, except for cycle 11 where a voltage of about 4 V was employed.

"Reference experiments on the ground (by turning the cell upside down) confirmed earlier observations of a very significant stratification under conditions where a normal fluid mixture would have been homogeneous." (7, p. 150)

Reportedly, unexpected results, related to the unusual properties of sodium chloride at its critical point, were observed. Only the last objective was achieved because solid NaCl precipitated just prior to launch (in the pre-equilibrium stage) between the electrodes. During the mission, NaCl re-dissolution dominated the conductance behavior. "During the flight, a microgravity related increase in solution resistance resulting from salt redissolution were observed." (7, p. 150) It was noted that the salt re-dissolution rate was 3-5 times faster than that on Earth.

<Note: Although it was not specifically stated, it appears that the test equipment performed as expected.>

Key Words: Critical Point Phenomena, Phase Transition, Two-Phase System, Binary Systems, Vapor/Liquid Transition, Sedimentation, Aqueous Solutions, Ionic Solution, Salt Solution, Compressible Solutions, Electrodes, Sedimentation, Conductance, Dissolution, Precipitation, Autoclave, Baffles, Corrosion, Impurities, Contained Fluids

Number of Samples: one (measurements taken during 12 cycles)
Sample Materials: supercritical aqueous sodium chloride (salt) solutions
(0.001 M solution of NaCl)
(Na*Cl*)
Container Materials: Synthetic sapphire tube with both endcaps made from stainless steel. A PtRh electrode also appears to have been in contact with the solution.

Experiment/Material Applications:
Water and steam provide the working fluid for virtually all large power stations. Ionic impurities can cause serious corrosion problems in several areas, such as turbines, where they can concentrate. The experiment was designed to facilitate the modeling water and steam behavior.

References/Applicable Publications:
(1) Turner, D. J.: Ionic Solutions Near the Critical Point of Water. In TEXUS 11/12 Abschlussbericht 1985, German Publication, pp. 66-68. (post-flight)

(2) Input received from Principal Investigator D. J. Turner, September 1989.

(3) Turner, D. J.: Electrolyte Solutions Near the Critical Point of Water: A Review and Proposal for Studies Under Microgravity Conditions. In ESA 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 393-400. (preflight)

(4) Turner, D. J.: Electrolyte Solutions Near the Critical Point of Water. In 6th European Symposium on Material Sciences Under Microgravity Conditions. Bordeaux, France, December 2-5, 1986, ESA 256, pp. 125-129. (post-flight TEXUS 10 and 12)

(5) Turner, D. J.: Salt Solutions in Supercritical Water. J. Chem. Soc., Faraday Trans. 1, 1988, 84(8), pp. 2683-2695. (post-flight)

(6) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near Critical Phenomena. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 222-256. (this experiment discussed on page 251; post-flight)

(7) Ionic Solutions Near the Critical Point of Water. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 150-153. (post-flight)

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Experiment Origin: Great Britain
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-9: conductance autoclave (modified post TEXUS 10 to ensure that solution contacted only non-corrosive materials). The heating profile was changed and the solution density increased to avoid the salt precipitation problem of TEXUS 10.
Builder of Processing Facility: Measurement and control facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany; autoclave: C.E.R.L., Leatherhead, Great Britain

Experiment:
Ionic Solutions Near the Critical Point of Water

This TEXUS 12 experiment was the second in a series of investigations designed by Turner to study ionic solutions near the critical point under low-gravity conditions (see Turner, TEXUS 10). The TEXUS 12 experiment was very similar to the TEXUS 10 experiment and had identical objectives.

The sodium chloride solution, contained within a thick-walled sapphire tube, was configured within a conductance autoclave. Pt-Rh endcaps now served as electrodes. Thus, the autoclave was modified so that solution contacted only non-corrosive materials. The heating rate was slowed and solution density was increased to avoid the salt precipitation problem of TEXUS 10.

Prior to the rocket launch, the temperature of the autoclave was ramped up to a temperature of 373 ^{o}C in 30 minutes. After this, the test temperature was slowly approached and held during the 2-hour period immediately before launch.

"The measurements of resistance and capacitance were made basically as in TEXUS 10, except that the high voltage (0.75 V) measurements were made during the ninth of the twelve, half minute measuring periods. The temperature of the solution was 377.5... [+/-] 0.5 ^{o}C and its concentration, as before, was 10^{-3} m. As previously, the autoclave was mounted so that the prelaunch resistance measurements were made at the bottom of the

cell. The solution resistance increased significantly in microgravity...." (7, p. 154)

Reportedly, all three objectives, as before stated (TEXUS 10), were achieved. Results indicated that destratification of the solution takes considerably longer than the 6 minutes of low gravity available during the rocket flight. "...To provide data suitable for thoroughly testing theories of compressible solutions, studies in orbiting craft will be required." (4, p. 129)

Key Words: Critical Point Phenomena, Phase Transition, Two-Phase System, Binary Systems, Vapor/Liquid Transition, Sedimentation, Aqueous Solutions, Ionic Solution, Salt Solution, Compressible Solutions, Electrodes, Sedimentation, Conductance, Autoclave, Baffles, Corrosion, Impurities, Contained Fluids

Number of Samples: one
Sample Materials: Test fluid: supercritical aqueous sodium cloride (salt) solution
(Na*Cl*)
Container Materials: synthetic sapphire tube with endcaps of a platinum-rhodium alloy
(Pt*Rh*)

Experiment/Material Applications:
Water and steam provide the working fluid for virtually all large power stations. Ionic impurities can cause serious corrosion problems in regions, such as turbines, where they can concentrate. Modeling their behavior is needed. This work attempted to facilitate this.

References/Applicable Publications:
(1) Turner, D. J.: Ionic Solutions Near the Critical Point of Water. In Texus 11/12 AbschluBbericht 1985, German Publication, pp. 66-68. (post-flight)

(2) Input received from Principal Investigator D. J. Turner, September 1989.

(3) Turner, D. J.: Electrolyte Solutions Near the Critical Point of Water: A Review and Proposal for Studies Under Microgravity Conditions. In ESA 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 393-400. (appears to be a precursor; preflight)

(4) Turner, D. J.: Electrolyte Solutions Near the Critical Point of Water. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5 1986, ESA 256, pp. 125-129. (post-flight TEXUS 10 and 12)

(5) Turner, D. J.: Salt Solutions in Supercritical Water. J. Chem. Soc., Faraday Trans. 1, 1988, 84(8), pp. 2683-2695. (post-flight)

(6) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near-Critical Phenomena. In Fluid Sciences and Materials Science in Space. Edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 222-256. (this experiment discussed on page 251; post-flight)

(7) Ionic Solutions Near the Critical Point of Water. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 154-155. (post-flight)

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Co-Investigator(s): Guenoun, P. (2), Perrot, F. (3)
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Experiment Origin: France
Mission: TEXUS 11
Launch Date/Expt. Date: April 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-10: Critical Point Facility (The TEM 06-10 module was completely redesigned for this experiment.)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Phase Separation in Binary Fluids: Spinodal Decomposition

The thermal quench of a binary fluid from its homogeneous to diphasic state produces either spinodal decomposition or nucleation. The resulting state is partly dependent on the convective forces present. Suppression of convection in such a process requires either a reduction of the gravity level acting on the system or a reduction of the density difference of the diphasic state.

This experiment was the first in a series of investigations designed by Beysens et al. to study the spinodal decomposition of a binary fluid. The objective of the research was to compare the ground-based phase separation of a two-component isodensity system (partially deuterated cyclohexane, methanol) to the low-gravity phase separation of an equivalent non-isodensity mixture (deuterated cyclohexane, methanol). The two experiments were performed under the same thermal conditions. Mixing of the components was achieved ultrasonically. Images of the phase separation were recorded by a 16 mm camera and subsequently subjected to computer image analysis.

Reportedly, a thermal stabilization within 0.1 mK was achieved around Tc = 45 ^{o}C, the critical temperature of the mixture. During the 6 minutes of low gravity available in the sounding rocket experiment, it was expected that the phase separation would reach a macroscopic level as the quench from the one-phase region (Tc + 5 mK) to the two-phase region (Tc - 10 mK) took place. The expected macroscopic spinodal decomposition was not observed, however, because the growth of the domains in the phase separating fluids was much slower than had been foreseen. "Nothing but the vapour bubble could be seen in the flight cell

even 6 mn after the quench, whereas macroscopic spinodal structures were clearly visible in the ground cell. The behaviour of the mixtures appeared very different although convection flows were essentially suppressed in both cases." (9, p. 156)

Possible explanations for the unexpected results were sought. A slight difference in composition (resulting in a high change in growth rate) or interface instabilities (possibly enhanced in low gravity) were suspected responsible. A second experiment (TEXUS 13, April 1986) was reportedly devoted to examining these issues.

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Isodensity Systems, Phase Stability, Binary Systems, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Interface Stability, Interface Physics, Nucleation, Spinodal Decomposition, Density Difference, Critical Density, Composition Distribution, Ultrasonic Mixing, Bubbles, Quench Process, Thermal Equilibrium, Contained Fluids

Number of Samples: one cell
Sample Materials: Space experiment: non-isodensity mixture of deuterated cyclohexane and methanol; Ground experiment: isodensity mixture of cyclohexane, deuterated cyclohexane and methanol.
Container Materials: fused quartz

Experiment/Material Applications:
During ground-based studies of the growth of domains in phase separating fluids, gravity effects can be removed by the use of mixtures whose densities can be precisely adjusted by partial deuteration. This investigation sought to characterize the mechanisms which drive such domain growth in space.

References/Applicable Publications:
(1) Beysens, F., Guenoun, P., and Perrot, G: Phase Separation of Binary Fluids Near its Critical Point. In Texus 11/12, Abschulussbericht 1985, German publication, pp. 43-45. (post-flight)

(2) Houessou, C., Guenoun, P., Gastaud, R., Perrot, F., and Beysens, D.: Critical Behavior of the Binary Fluids Cyclohexane-methanol, Deuterated Cyclohexane-methanol and Their Isodensity Mixture: Application to Microgravity Simulations and Wetting Phenomena. Physical Review A, Vol. 32, Number 3, September 1985, pp. 1118-1833. (does not appear to have space results)

(3) Beysens, D.: Critical Point Phenomena in Fluids. In ESA 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, June 1983, pp. 367-376. (no space results)

(4) Beysens, D., Guenoun, P., and Perrot, F.: Phase Separation in Microgravity of Binary Fluids Near A Critical Point. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA-256, pp. 139-144. (TEXUS 11, 13)

(5) Beysens, D.: Stability of Critical Fluid Mixtures: Experimental Simulation of Microgravity. Acta Astronautica, 12, 141 (1985).

(6) Beysens, D., Guenoun, P., and Perrot, F.: Experimental Study of Phase Separation in a Critical Mixture Under Microgravity. Workshop on Composites, Both Artificial and In Situ in the Earth's and the Space Laboratory (Grenoble, 1985), p. 178.

(7) Beysens, D.: Critical Phenomena. Materials Science in Space, edited by B. Feuerbacher, H. Hamacher, and R. J. Neumann (Springer, 1986), p. 191.

(8) Input received from Principal Investigator D. Beysens, June 1988.

(9) Phase Separation of a Binary Fluid Near Its Critical Point. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 156-157. (post-flight)

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Co-Investigator(s): Guenoun, P. (2), Perrot, F. (3)
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Experiment Origin: France
Mission: TEXUS 13
Launch Date/Expt. Date: April 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-10: Critical Point Facility (including a double thermostat and an ultrasonic oscillator (for swift homogenization)). TEM 06-10 was originally developed for TEXUS 11/12.
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Phase Separation in Binary Fluids: Spinodal Decomposition

This experiment was the second in a series of investigations designed by Beysens et al. to study the spinodal decomposition of a binary fluid (see Beysens, TEXUS 11). Slow growth rates of the domains in phase separating fluids, encountered in the non-isodensity system deuterated cyclohexane and methanol during phase separation on TEXUS 11, prompted this investigation.

During TEXUS 13, an isodensity mixture of partially deuterated cyclohexane and methanol was subjected to the same conditions as the non-isodensity system flown on TEXUS 11. A CCD video camera with telemetry was added to the experiment module.

Results of the TEXUS 13 experiments were compared to similar isodensity experiments performed in the ground-based laboratory, enabling specific effects, attributed to the low-gravity conditions, to be evidenced.

Post-flight analysis illustrated that essentially the quenched flight sample behaved qualitatively, as did the ground sample which also employed the isodensity mixture. Both rocket and Earth samples developed microscopic structures which covered the size of the sample over time. "The pattern is highly interconnected. The final state corresponds to one phase (M-rich) wetting of the walls of the container and surrounding the other phase." (1, p. 139) A bubble in the flight sample distinguished it from the Earth sample.

It was concluded that "...the slow growth rate observed in TEXUS 11 was due to a minor (1%) deviation from the critical concentration; this growth could be reduced on the ground with the isodensity system.... [In addition,] The gravity effects, at least in the two-phase critical region and excepting for the very late stages, can indeed be removed with precision in special ground-based systems. Here they authorize valid simulations of phase transitions in space." (11)

<Note: Several of the references listed below were not available at the time this experiment summary was prepared.>

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Isodensity Systems, Phase Stability, Binary Systems, Buoyancy-Driven Convection, Interface Stability, Interface Physics, Nucleation, Spinodal Decomposition, Density Difference, Composition Distribution, Ultrasonic Mixing, Bubbles, Quench Process, Wetting of Container, Contained Fluids

Number of Samples: one cell
Sample Materials: A mixture of three components: cyclohexane (C), deueterated Cyclohexane (C*) and methanol (M) (space and ground experiments)
Container Materials: fused quartz
(Si*O*)

Experiment/Material Applications:
See Beysens, TEXUS 11

References/Applicable Publications:
(1) Beysens, D., Guenoun, P., and Perrot, F.: Phase Separation in Microgravity of Binary Fluids Near a Critical Point. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA-SP-256, pp. 139-144. (TEXUS 11, 13)

(2) Beysens, D., Guenoun, P., and Perrot, F.: Phase Separation in Microgravity of Binary Fluids Near a Critical Point. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 46-52. (post-flight)

(3) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near Critical Phenomena. In Fluid Sciences and Materials Science in Space: A European Perspective, Edited by H. U. Walter (Springer-Verlag, 1987), pp. 221-256.

(4) Guenoun, P., Gastaud, R., Perrot, F., and Beysens, D.: Spinodal Decomposition Patterns of an Isodensity Critical Binary Fluid: Direct Visualization and Scattering Analyses. Phys. Rev. A36, 4876 (1987).

(5) Beysens, D., Guenoun, P., and Perrot, F.: Phase Separation of Critical Binary Fluids Under Microgravity. Comparison with Matched Density Conditions. Phys. Rev. A (1988). <Note: Additional information concerning this document was not provided.>

(6) Guenoun, P.: Instabilités des mélanges de fluids. Inflence des forces de pesanteur et de mouillage. Thése, April 1987. (Paris VI)

(7) Perrot, F., Guenoun, P., and Beysens: Phase Separation of Binary Fluids Near a Critical Point Under Microgravity. Proceedings of a NATO Advanced Research Workshop on Hydrodynamics: Interfacial Phenomena, Huelva, July 1986, eds., M. G. Velarde and B. Nichols (Plenum Press, 1988), p. 171.

(8) Beysens, D., Guenoun, P., and Perrot, F.: Phase Separation in Fluids in the Absence of Gravity Effects. International Symposium on "Dynamics of Ordering Process in Condensed Matter," Kyoto, August 27-30, 1987.

(9) Experiment-Modul TEM 06-10. In TEXUS 13-16 Abschussbericht, BMFT/DFVLR, p. 45. (in German; post-flight, processing facility)

(10) Beysens, D., Guenoun, P., and Perrot, F.: Phase Separation in Microgravity of Binary Fluids Near a Critical Point. In TEXUS 13-16 Abschlussbericht, BMFT/DFVLR, p. 45. (post-flight)

(11) Input received from Principal Investigator D. Beysens, June 1988.

(12) Phase Separation in Microgravity of Binary Fluids Near the Critical Point. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 158-161. (post-flight)

Additional references can be found under Beysens, TEXUS 11.

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Co-Investigator(s): Unknown
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Experiment Origin: France or Belgium
Mission: Unknown, possibly STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: Unclear
Payload Type: Unknown
Processing Facility: Unknown
Builder of Processing Facility: Unknown

Experiment:
Phase Separation of a Critical Mixture of Isobutyric Acid and Water

<Note: Although Reference (1) (below) briefly introduced and described this experiment, no further details concerning its objectives, setup, or results could be located at this time. The following paragraph represents nearly the entire experiment summary as reported in Reference (1):>

"This demonstration was performed in a Space Shuttle in June 1985.... The isobutyric acid-water system was chosen because it meets the safety requirements for the manned flight and also because its critical temperature, Tc = 30 ^{o}C, can be easily reached. The isobutyric-rich phase was coloured green by traces of malachite. The experiment consisted of first, the observation of wetting layers at room temperature, where the system was in the two-phase region. Then the cell was heated by hand contact and stirred. The phase separation process could be observed by letting the temperature of the cell return to room temperature. Very long equilibrium times were observed. As expected, the wetting layers were found to be macroscopic." (1, p. 246)

Key Words: Critical Point Phenomena, Phase Separation, Two-Phase System, Binary Systems, Wetting, Stirring of Components

Number of Samples: unknown

Sample Materials: Isobutyric acid-water system.
The isobutyric-rich phase was colored green using traces of malachite.
Container Materials: unknown

Experiment/Material Applications:
Unknown

References/Applicable Publications:
(1) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near Critical Phenomena. In Fluid Sciences and Materials Science in Space, edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 222-256. (post-flight; this experiment is specifically described on pages 246-247.)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Process Chamber Double Rack
Processing Facility: Holographic Optical Laboratory (HOLOP) (Holographic Interferometer)
Builder of Processing Facility: Designed and built by the Institute for Space Simulation, DFVLR, Cologne, Germany

Experiment:
Density Distributions and Phase Separation in Fluids at the Critical Point (PK-HOL-02)

During terrestrial experiments, gravity-driven forces induce inhomogeneities in a fluid as it approaches the gas/liquid critical point. During similar space experiments, such gravity-driven forces are reduced, and a homogeneous density distribution is expected to be maintained near the transition point. If a homogeneous distribution is attained, closer examination of important critical-point fluid/vapor characteristics should be possible.

This Spacelab D1 experiment was the first in a series of experiments designed by Klein to study the phase separation of fluids under low-gravity conditions. During the D1 mission, two-phase sulfur hexafluoride (SF_6), contained within a glass capillary, was heated to approximately 3 K above its critical temperature, Tc, and then cooled slowly. Reportedly, "The capillary was contained in a copper thermostat with viewing windows for illumination and with 4 thermistors for temperature and control." (6, p. 196) Double exposure holographic measurements, made using the D1 HOLOP interferometer, allowed analysis of the fluid density distribution.

HOLOP images revealed that appreciable density inhomogeneities were present in the flight sample. Results indicated that the inhomogeneities "...are sustained by adiabatic temperature gradients and thus may survive phase transition. Near the critical point, local mass differences inside the sample swell as a response to the adiabatic temperature gradients via thermal ex-

pansion. In this way, initially minor inhomogeneities may become as large as those caused by 1 g on earth." (1, p. 374).

Investigators concluded that a low-gravity condition "...is in no way sufficient for establishing homogeneity of a fluid of overall critical density when heating through the critical temperature from a two-phase region to a the single phase region." (1, p. 374)

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Binary Systems, Capillaries, Buoyancy-Driven Convection, Density Distribution, Homogeneity, Vapor/Liquid Transition, Critical Density, Thermal Gradient, Thermal Expansion, Holography, Interferometric Measurement, Contained Fluids

Number of Samples: one
Sample Materials: Sulfur hexafluoride (SF_6): overall critical density = 0.73 $g*cm^{-3}$; critical temperature = 318.69 K (S*F*)
Container Materials: glass capillary

Experiment/Material Applications:
An increase in density homogeneity of a sample at the liquid/vapor critical point was anticipated in the low-gravity environment. (See **Experiment** summary (above).)

References/Applicable Publications:
(1) Klein, H. and Wanders, K.: Density Distribution Near Gas/Liquid Critical Points Under Reduced Gravity. Natur wissenschaften, 73. Jahrgang Heft 7, July 1986, pp. 374-375. (post-flight)

(2) Klein, H. and Wanders, K.: Density Distribution and Phase Transition Near Gas/Liquid Critical Points Under Reduced Gravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 30.

(3) Klein, H. and Wanders, K.: Holographic Interferometry Near Gas-Liquid Critical Points, Ground Based Study of a D1 Experiment. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 323-325. (preflight)

(4) Klein, H. and Wanders, K.: Density Distribution and Phase Separation in Fluids at the Critical Point. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 86.

(5) Wanders, K., Klein, H., Bewersdorff, A., and Neuhaus, D.: Holographic Experiments: Results of Spacelab D 1. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, pp. 105-107. (post-flight)

(6) Klein, H. and Wanders, K.: Density Distribution and Phase Transition Near Gas/Liquid Critical Points Under Reduced Gravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 196-198. (post-flight)

(7) Beysens, D., Straub, J., and Turner, D. J.: Phase Transitions and Near-Critical Phenomena. In Fluid Sciences and Materials Science in Space, edited by H. U. Walter, Springer Verlag, Berlin, 1987, pp. 222-256. (D1 experiment is discussed on p. 246; post-flight)

(8) Hammacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements on D1)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 17
Launch Date/Expt. Date: May 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-14
Builder of Processing Facility: Unknown

Experiment:
Spinodal Decomposition

"Phase separation close to the critical point evolves from the unstable region of the phase diagram. [Subsequent] Phase growth under unstable conditions, particularly during the early... stages is envisaged as occurring by spinodal decomposition." (1, p. 369) Spinodal decomposition has been observed in several material systems: (1) metallic alloys, (2) inorganic glasses, (3) small molecule liquid mixtures, (4) polymer blends, and (5) solutions of macromolecules in small-molecule solvents.

During the terrestrial study of spinodal decomposition in binary liquid mixtures, characteristic laser light scattering patterns have been observed. These patterns are typically rings with a granular appearance (see Reference (1) for a detailed discussion). "Analogies between phase transitions suggest that the spinodal decomposition of a single component fluid can be studied by means of light-scattering techniques in the same way as the spinodal decomposition of binary liquid mixtures. Density fluctuations in a single component fluid act on the light beam in the same way as concentration fluctuations in a binary mixture thus yielding a ring of maximum scattered-light intensity." (2, p. 162)

"The samples with critical overall density are transferred by a rapid change of temperature (temperature quench) from a state of the single phase region above the critical temeprature into a state of the thermodynamically unstable region below the critical temperature. The ensuing decomposition of the system into two coexisiting phases proceeds by enhancement of the order parameter fluctuations.... [Specific parameter fluctuations cause] the observed ring shaped maximum of scattered light intensity...." (1, p. 369)

Terrestrial experiments designed to study spinodal decomposition of single-component fludis are complicated by gravity induced forces which result in (1) density inhomogeneities near the gas/liquid critical point and/or (2) sedimentation and buoyancy of the emerging gaseous or liquid phases.

Ground-based experiments with the single-component fluid sulfur hexafluoride, SF_6 (critical density = 0.74 g/cm^3, Tc = 318.7 K), produced high-quality scattering rings with a lifetime of approximately 1.5 seconds. During the first 0.5 second of the ring's life, the radius was nearly constant and the maximum intensity of the scattered light increased exponentially with time. "The measurements revealed that, due to continuous quenching conditions, the spinodal decomposition phenomena observed can be formally described by the current linear [Cahn] theory. However, the comparison with what is observed with binary liquid mixtures led to the assumption that the 'spinodal' rings should be stable for a longer time under instantaneous quenching, reduced multiple scattering, and microgravity conditions." (2, p. 162)

This TEXUS 17 experiment was the second in a series of investigations designed by Klein to study the phase separation of fluids under low-gravity conditions (see Klein, Spacelab D1).

<Note: Discussions which specifically describe the flight experimental apparatus and procedures were not provided in the available publications. Reference (1) describes the apparatus and procedures used during ground-based experiments and then states that "Experiments under earth bound conditions as well as under reduced gravity conditions... have been carried out with the same apparatus and quenching protocol." (1, p. 370) See Reference (1) for further details. Reference (2) also makes a similar statement about the similarity between ground and flight experiments.>

The TEXUS 17 experiment was performed in the TEM 06-14 experimental module. It is suspected (via Reference (1)) that (1) the module contained a 12.4 mm diameter cylindrical copper cell (with sapphire windows) filled with SF_6, (2) the cell was equipped with a magnetic stirring bar and centrally located thermistor, (3) the temperature within the cell could be controlled to within 0.3 mK, (4) cell liquid quench from the initial single-phase region to the final two-phase region was activated via Peltier elements, (5) a He-Ne laser beam was passed through the liquid sample, and (6) scattered intensities at small scattering angles were recorded photographically and on video tape (50 pictures/s).

During the TEXUS 17 experiment, it is suspected (via References (1) and (2)) that (a) the test fluid was heated to Tc + 6.0 +/- 0.5 mK, (b) the test fluid was stirred vigorously to insure den-

sity homogeneity, (c) the stirrer was shut off and during the following 20 seconds, fluid turbulences were allowed to diminish, and (4) the test fluid was cooled at a rate of 2 mK/s (as measured at the container wall adjacent to the liquid) to Tc - 3.0 +/- 0.5 mK.

It was reported that the results from the flight experiment were similar to those obtained during ground-based studies. This indicated that gravity did not influence the spinodal decomposition process during the short time available on the rocket flight. "It was concluded that spinodal decomposition is much more affected by a finite quenching rate in a single-component fluid than in a binary mixture. This is due to the fact that the relaxation time of the density fluctuations in the former is smaller than the relaxation time of the concentration fluctuations in the latter." (2, p. 162)

No further conclusions related specifically to the reduced gravity experiment could be located at this time.

<Note: Reference (3) (as listed below) was not available to aid in the preparation of this experiment summary.>

Key Words: Critical Point Phenomena, Phase Separation, Phase Transition, Two-Phase System, Binary Systems, Stirring of Components, Spinodal Decomposition, Buoyancy-Driven Convection, Sedimentation, Density Distribution, Homogeneity, Turbulent Flow, Vapor/Liquid Transition, Critical Density, Quench Process, Cooling Rate, Laser Light Scattering, Contained Fluids

Number of Samples: one
Sample Materials: sulfur hexafluoride, SF_6
Container Materials: Unclear, appears to have been a copper experiment cell with sapphire windows.
(Cu*)

Experiment/Material Applications:
See **Experiment** section (above).

References/Applicable Publications:

(1) Klein, H., Schmitz, G., and Woermann, D.: Early Stages of Phase Separation of a Single-Component Fluid at the Critical Point. In Proceedings of the Seventh European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-295, January 1990, pp. 369-371. (post-flight)

(2) Spinodal Decomposition. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 162-163. (post-flight)

(3) Klein, H., Schmitz, G., and Woermann, D.: Spinodal Decomposition in a Single-Component Fluid. Physics Letters A, Vol. 136, Nos. 1 and 2, 73-76.

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CHAPTER 8

CRYSTAL GROWTH FROM SOLUTION

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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: November 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: Crystalline materials sealed in a food can
Builder of Processing Facility: (1) University of Alabama, Tuscaloosa, Alabama, (2) Marshall Space Flight Center, Huntsville, Alabama, and (3) Johnson Space Center, Houston, Texas

Experiment:
Rochelle Salt Growth (SD33(TV105))

Research has illustrated that crystals grown from solution are adversely affected by gravity-induced convection.

This Skylab SL-4 science demonstration was designed to study low-gravity solution crystal growth. The specific objective of the experiment was to study the growth when buoyancy-driven convective flows were absent in the depleted solutal layer around the growing crystal.

During the Skylab SL-4 experiment, a food can was filled with the following three materials: (1) a saturated Rochelle salt solution (saturation at 25 ^{o}C), (2) Rochelle salt powder, and (3) a 26 gram Rochelle salt seed crystal. The can was equipped with a see-through membrane under the pull-top lid. After removing the lid, the can was heated to 70 ^{o}C. Heating continued until approximately three quarters of the seed crystal were dissolved. The can was then removed from the heat, wrapped in three towels, and stored. As the can slowly cooled over a period of 2 days, the seed crystal began to regrow. The towels were removed and the crystals were photographed using a 35 mm camera. Two weeks later the can was opened and the seed crystal was removed from the can and examined.

During post-flight debriefings, the astronaut who performed the experiment "...described the solution as 'slushy' and the self-nucleated crystallites as 'mica-like'." (7, p. 16) This condition indicated the solution was still supersaturated when the can was opened.

Inspection of the recovered material revealed the presence of three large crystals: (1) the first having a mass 8.73 grams, (2) the second having a mass of 2.82 grams, and (3) the third having a mass of 0.50 grams. In addition, 16 smaller crystals were obtained. Photographs obtained during the mission indicated that "The crystal originally precipitated in the [Skylab] cabin is believed to be one single crystal.... Thus, most of the other small crystals, if not all of them, probably appeared when the big crystal was broken. The breaking of the large crystal probably occurred during re-entry or splash-down of the module.

"The crystal grown on board Skylab-4 is actually a collection of at least five single crystals.... One very unusual thing about this collection of crystals is that the a, b, and c crystal axes of all the component crystals are parallel to each other." (5, p. 3)

Micro- and macroscopic examination of the crystals revealed many defects, although some parts of the crystals were almost completely free from defects and appeared to be "optically good." Almost of all the crystals returned from Skylab contained defects which were parallel to the c axis of the crystal. Typically, the defects were tubular (e.g., about 4 mm long in the 2.82 gram crystal). There were a few spheres of trapped solution in the tubular cavities but they were very small.

Measurements of the ferroelectric quality of the crystals were made by determination of the S-value, calculated from hysteresis curves (high S-values indicate uniform domain structure and better ferroelectric properties). Because of the fragility of the space-grown crystals, however, slices could not be cut and the entire crystals were used for the analysis. (It was reported that one of the goals of further research was to determine an acceptable slicing method.) Reportedly, the S-value for the Skylab crystal was 1.36, and the S-value for an Earth-grown material was 2.69. It was concluded that the presence of unevenly distributed cavities within the space crystal resulted in the lower S-value. Accurate S-values can only be obtained from thin slices of the crystal and, therefore, a better picture of the ferroelectric property was expected to be attained with the development of an appropriate slicing method.

Key Words: Crystal Growth From Solution, Salt Solution, Seed Crystals, Single Crystals, Supersaturation, Precipitation, Boundary Layer, Passive Cooling, Solid/Liquid Interface, Surface Morphology, Crystalline Defects, Defect Density, Crystalline Structure, Nucleation, Cavity, Powders, Coated Surfaces, Buoyancy-Driven Convection, Contained Fluids, Acceleration Effects, Payload Survivability, Vehicle Re-entry Forces/Vibration

Number of Samples: one
Sample Materials: Rochelle salt solution, Rochelle salt powder, Rochelle salt seed crystal, and water
Container Materials: Skylab food can. <Note: The Principal Investigator reported that he thought the 3-inch diameter, 1-inch deep can was comprised of Teflon™-coated aluminum.>

Experiment/Material Applications:
This investigation was performed to determine the feasibility of producing high quality materials during future low-gravity crystal growth initiatives.

Rochelle salt was chosen for the experiment because large crystals of the substance are easily grown.

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(1) TV105-Rochelle Salt Growth. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-21 - 7-23. (post-flight)

(2) Input received from Experiment Investigator, September 1988 and July 1993.

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(7) Bannister, T. C.: Skylab III and IV Science Demonstrations. Preliminary Report, NASA TM X-64835, March 1974, pp. 15-17. (post-flight)

(8) Naumann, R. J. and Mason, D.: Rochelle Salt Growth. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 44. (post-flight)

(9) Rochelle Salt Growth (DS33-TV105). MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-92.

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Co-Investigator(s): Unknown
Affiliation(s): (1) During Skylab: Lockheed Missiles and Space Company, Inc., Huntsville, Alabama, Currently: Faratech, Inc., Huntsville, Alabama; (2) National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama

Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: January 1974 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: Lexan™ polycarbonate vial equipped with copper wire for silver deposition
Builder of Processing Facility: Unknown

Experiment:
Deposition of Silver Crystals (SD21 TV106)

The objective of this Skylab science demonstration was to determine the effect of the space environment on the production of silver crystals by an electrochemical reaction.

During the SL-4 mission, crystals were allowed to grow on the surface of a coiled, insulated wire held in a Lexan™ tube (10 ml volume). The tube contained 9.5 ml silver nitrate solution (5% by weight). (The wire insulation had been scarred by a Skylab astronaut (using a pocket knife) to provide nucleation sites for the growth of silver crystals.) Prior to insertion of the coiled wire, the tube was placed in the Skylab centrifuge and rotated to settle the solution. After centrifuging the tube/solution, the wire was inserted into the tube which was then sealed.

The crystals which formed on the wire were to be photographed by a 35 mm camera after approximately 6, 24, and 76 hours of crystal growth. However, "...some unknown mishap occurred and no pictures developed on the exposed film." (5, p. 178)

Corresponding experiments were performed on Earth under (a) 1-g conditions and (b) 2 to 5-g conditions (using a centrifuge). <Note: It was assumed that the Earth-based experimental procedures were similar to the space procedures, although this fact was not specifically stated.>

The astronaut described the crystals as long dendrites and that most of the crystal growth had occurred during the first 24 to 48 hours of the experiment. Crystal growth was almost complete by 72 hours after insertion of the copper wire.

Earth-based examination of the flight sample indicated that other than planned reactions had occurred in the tube. "Original solutions before insertion of copper wire contained approximately 3 x 10^7... [micrograms/liter]... Ag and O... [micrograms/liter]... Cu. If all of the silver had been replaced and no other reactions occurred, the copper concentration would have been about 6 x 10^6... [micrograms/liter].... This is not the only evidence for additional reactions. Small amounts of both blue and dark red crystals of unidentified materials are obtained in any ground electrochemical displacement of silver ions by copper, in addition to silver crystals. Such blue and red crystals were obtained also in the flight experiment. Further analysis of these colored crystals, however, were not undertaken." (5, p. 179) <Note: Discussions of the specific reactions which may have produced these blue and red crystals were not presented in the available published literature.>

Certain tendencies in habit formation, as a function of gravity, were discerned from the low-gravity and Earth experiments. These included:

(1) The low-g materials were more powdery and less coherent than the high-g crystals: the higher the gravity level, the more coherent.

(2) Examination of the low-g crystals indicated a more perfect crystalline form than the high-g materials: the higher the gravity level, the less perfect the resultant structure.

(3) The crystals grown at high gravity levels showed evidence of rapid growth from the edges and corners. <Note: This characteristic was not mentioned for the low-gravity material.>

(4) Curvature in the crystals caused by fluid flow is evident in the material produced under 1-g conditions. The low-g material contained no such curvature.

(5) It appeared that steps and ledges were more prevalent in the higher gravity crystals.

(6) As the gravity level increased, it appeared the amount of twinning also increased.

Other results from the ground-based experiments (1-g and higher-g) included:

(1) When dilute solutions were employed, dendrite streamers grew parallel to the gravity vector; when concentrated solutions were employed, streamers of chunky, imperfect crystals grew opposite to the gravity vector.

(2) Centrifuging dilute solutions increased coherency and size of dendrite streamers.

(3) Crystal streamers formed from concentrated solutions, and after encountering a free liquid/gas surface, spread out.

(4) As solution concentration increased, gas generation also increased.

It was also reported that the "...trends in crystalline morphologies observed in earth- and space-grown silver crystals almost exactly parallel those observed in earth- and space-grown germanium selenide (GeSe) and telluride (GeTe) crystals." (5, p. 181) (see Wiedemeier, Skylab SL-3 and Skylab SL-4 M556 experiments (Chapter 10).)

The above results led to the following conclusions:

(1) The observed differences between space- and Earth-grown crystals were attributed to the differences in the rate of electrochemical displacement of silver ions from a 5% aqueous solution by copper: under low-gravity conditions, the displacement rate tends to be diffusion controlled and at higher gravity levels, kinetically controlled.

(2) The downward and upward (relative to the gravity vector) growth of crystal streamers was attributed to gravity-driven convection. The spreading out of the streamers at the liquid/gas interface probably was due to surface-tension driven flow.

(3) Electrolysis under low-gravity conditions results in (a) dendritic crystals with better microcrystalline structures or (b) large single crystals with fewer defects than can be produced at higher gravity levels.

(4) It appears that the vapor transport growth of GeSe and GeTe (see Wiedemeier's Skylab SL-3 and SL-4 experiments (Chapter 10)) is affected by convection in the same way that is theorized for electrochemical deposition of silver crystals.

Key Words: Crystal Growth From Solution, Electrochemical Reaction, Reaction Kinetics, Aqueous Solutions, Electrodeposition, Electrolysis, Binary Systems, Ternary Systems, Single Crystals, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Surface Tension-Driven Convection, Growth Rate, Solid/Liquid Interface, Concentration Distribution, Gas Formation, Liquid/Gas Interface, Nucleation, Nucleation Sites, Dendrites, Dendritic Solidification, Dendritic Structure, Crystalline Structure, Sample Homogeneity, Crystal Morphology, Crystalline Defects, Twinning, Contained Fluids, Processing at High Gravity Levels, Centrifuge, Contamination Source, Photographic Difficulties

Number of Samples: one
Sample Materials: 22-gauge copper wire placed in silver nitrate solution
(Ag*Ni*, Cu*)
Container Materials: Lexan^{TM} plastic

Experiment/Material Applications:
No discussion of the experiment/material application could be located in the published literature. However, it appears that the experiment sought to determine if purely diffusive fluid flow (rather than diffusion plus convection) would occur in the chosen crystal growth system.

References/Applicable Publications:
(1) Grodzka, P. G., Facemire, B. R., Johnston, M. H., and Gates, D. W.: Electrochemical Deposition of Silver Crystals Aboard Skylab IV. NASA TN D-8277, July 1976, 43 pp. (post-flight)

(2) Grodzka, P.: Three Model Space Experiments on Chemical Reactions. In the Marshall Space Flight Center Bioprocessing in Space, pp. 67-76. (post-flight) <Note: Document number unclear.>

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(5) Grodzka, P. G., Facemire, B. R., Johnston, M. H., and Gates, D. W.: Electrochemical Deposition of Silver Crystals Aboard Skylab IV. Journal of Crystal Growth, Vol. 35, 1976, pp. 177-184. (post-flight)

(6) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(7) Bannister, T. C.: Skylab III and IV Science Demonstrations Preliminary Report. NASA TM X-64835, March 1974, pp. 17-20. (post-flight)

(8) Naumann, R. J. and Mason, D.: Deposition of Silver Crystals. In Summaries of Early Materials Processing in Space Experiments, NASA TM 78240, August 1979, p. 45. (post-flight)

(9) Deposition of Silver Crystals. In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-90.

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Co-Investigator(s): None
Affiliation(s): (1) During ASTP: Rockwell International Science Center, Thousand Oaks, California, Currently: Retired

Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Manned Environment (mounted in a U4 locker in the command module)
Processing Facility: Six chemical reactors, each with three compartments separated by valves
Builder of Processing Facility: Science Center, Rockwell International Science Center, Thousand Oaks, California

Experiment:
Crystal Growth (MA-028)

The gel method of crystal growth involves two or more reactant solutions separated by a gel. The gel material is usually comprised of 95% or 96% water and 4% or 5% solids. During the crystal growth process, the solutions diffuse through the gel and react with each other to form insoluble crystals. The gel acts to (1) suppress gravity-driven convection and (2) support the growing crystal.

Gels can negatively affect crystal growth. For example, excessive nucleation can take place in the gel resulting in small crystals. Further, the crystal can become contaminated by gel constituents or impurities. In general, low-gravity processing of crystals was expected to eliminate the disadvantages and requirements of a gel medium while retaining the overall advantages of the gel technique.

This ASTP experiment was the first in a series of investigations designed by Lind and/or Nielsen et al. to study the solution growth of organic crystals under low-gravity conditions. The specific objective of the experiment was to determine the feasibility of growing single crystals by a technique in which two reactant solutions diffuse toward each other through a pure solvent. This method of crystal growth is closely related to growth by the gel method.

Prior to the flight, six experiment reactors were prepared to study three different crystal growth systems (two reactors for each system). The reactors were constructed of a clear Lexan™ polycarbonate resin and were equipped with silicone rubber gaskets and seals. Each reactor contained three chambers. The

chamber containing the solvent was sandwiched between two chambers, each of which contained a reactant solution. (The chambers varied in length.) Separating the chambers were porous, glass diaphragms. Prior to delivery to the launch site, the chambers were filled with the appropriate solutions.

During the ASTP mission three crystal growth reactions were studied. In the first reaction, calcium tartrate ($CaC_4H_4O_6$) was grown from aqueous solutions of $CaCl_2$ and $NaHC_4H_4O_6$. In the second reaction, calcium carbonate ($CaCO_3$) was grown from solutions of $CaCl_2$ and $(NH_4)_2CO_3$. In the third reaction, lead sulfide (PbS) was grown from solutions of $PbCl_2$ and CH_3CSNH_2. While under low-gravity conditions, the experiments were initiated by opening intercompartment valves and allowing the solutions to diffuse together. At the start of the experiment an astronaut photographed the six reactors with a 35 mm camera. During the following 116 hours the astronaut photographed the reactors once every 12 hours. (A framing device was used to properly position the camera.) The reactions were allowed to occur at cabin-ambient temperatures.

It was reported that the experiment operated as planned with no observed leakage of the fluids before or during the experiment.

Post-flight examination of the photographs and grown crystals indicated that "Crystals of about the expected size, quality, and number formed in all six reactors." (1, p. 558) It was noted that the temperature variation in the spacecraft, between 60 ^{o}F and 75 ^{o}F, was greater than expected.

Reportedly, the calcium tartrate crystals were the best obtained. This result was expected because these crystals are among the easiest to grow using the gel method.

The calcium tartrate reactors produced numerous prismatic crystals as large as 2 mm and plate-like crystals of up to 5 mm in length. Unlike gel-grown crystals, the plate-like crystals were larger and more numerous than the prismatic crystals. X-ray diffraction studies indicated that they were of good quality. Some crystals about 10 mm in length were also formed, but their quality was not as good as the shorter crystals.

Both calcium carbonate reactors produced numerous well-formed, clear rhombohedral crystals as long as 0.5 mm on edge. These crystals were similar in appearance to those grown on Earth and were large enough to exhibit birefringence. However, most of the crystals adhered to the polycarbonate walls of the reactor indicating that this material was not the best choice to demonstrate this reaction.

Reportedly, the two PbS experiments were not considered successful, although discrete single crystals as large as 0.1 mm were produced. The mixing and reaction process was not completed prior to re-entry. Even after return of the reactors, a fine precipitate continued to grow. This indicated that for this crystal system, a longer period of time or a higher temperature is required to obtain high quality crystals.

It was concluded that low-gravity processing resulted in crystals of approximately the same size, quality, and number as those obtained by the gel method on Earth (in the same length of time). "Nucleation was excessive in all six reactors, which indicates the need for considerable refinement of the experimental parameters to suppress nucleation and improve crystals size and quality. Precise temperature control and longer growth times are probably the two main improvements to be made." (1, p. 562)

Key Words: Crystal Growth From Solution, Reactant Solutions, Aqueous Solutions, Organic Crystals, Binary Systems, Ternary Systems, Insoluble Crystals, Single Crystals, Solvent, Diffusion, Double Diffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Liquid/Liquid Interface, Solid/Liquid Interface, Thermal Environment More Extreme Than Predicted, Crystal Morphology, Crystalline Structure, Nucleation, Platelike Structure, Birefringence, Wetting, Liquid Reservoir, Contained Fluids, Material Interaction With Containment Facility, Crucible Effects, Optics Applications, Semiconductors, Semiconductor Applications, Electronic Materials, Incomplete Sample Processing

Number of Samples: six
Sample Materials: (1) calcium tartrate, $CaC_4H_4O_6$, (2) calcium carbonate, $CaCO_3$, and (3) lead sulfide, PbS (See Reference (1) for reactant solutions.)
(Ca*C*H*O*, Ca*C*O*, Pb*S*)
Container Materials: Lexan™ polycarbonate resin

Experiment/Material Applications:
Calcium tartrate, calcium carbonate, and lead sulfide crystals were chosen this experiment because:

(1) These crystals are easily grown in gel systems.
(2) Data on these crystals, grown on Earth in gel systems, are readily available for comparison.
(3) Calcium tartrate has been especially important for studies in gel systems.
(4) Calcium carbonate and lead sulfide have technological application and importance (optical devices ($CaCO_3$), light detectors (PbS).
(5) Calcium carbonate is typically contaminated by gel constituents.

It was suspected that the crystal growth of calcium carbonate, therefore, may be improved without the use of gels.

References/Applicable Publications:

(1) Lind, M. D.: Crystal Growth. In Apollo-Soyuz Test Project Summary Science Report, NASA SP-412, Vol. I, pp. 555-562. (post-flight)

(2) Lind, M. D.: Crystal Growth from Solutions in Low Gravity. AIAA 15th Aerospace Sciences Meeting, Los Angeles, California, January 24-26, 1977, 8 pp. (post-flight)

(3) Lind, M. D.: Experiment MA-028 Crystal Growth--Low Gravity Manufacturing of Single Crystals from ASTP. Final Report, May 1974-August 31, 1976, NASA Report NASA CR-150984, 22 pp.

(4) Nielsen, K. F. and Lind, M. D.: Solution Crystal Growth on the Apollo-Soyuz, the Spacelab, the LDEF, and the EURECA Missions. In Proceedings of the Fifteenth International Symposium on Space Technology and Science, Tokyo, 1986, pp. 2111-2116.

(5) Wiedemeier, H.: Crystal Growth in Micro-Gravity, An Overview. In Applications of Space Flight in Materials Science and Technology, proceedings of a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, pp. 25-39, issued September 1988.

(6) Naumann, R. J. and Mason, E. D.: Crystal Growth. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 54-55. (post-flight)

(7) Lind, M. D.: Crystal Growth from Solutions in Low Gravity. AIAA Journal, Vol. 16, No. 5, May 1978, pp. 458-462. (post-flight)

(8) Input received from Principal Investigator M. D. Lind, August 1993.

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Principal Investigator(s): Lind, M. D. (1), Nielsen, K. F. (2)
Co-Investigator(s): None
Affiliation(s): (1) During STS-013: Rockwell International Science Center, Thousand Oaks, California, Currently: Retired; (2) Physics Laboratory, Technical University of Denmark, Lyngby, Denmark

Experiment Origin: USA/Denmark
Mission: Launched: STS Launch #11, STS-013 (STS 41-C, Challenger); Returned: STS-032 (Columbia)
Launch Date/Expt. Date: April 1984. The experiment, which was on the LDEF free-flying facility, orbited the Earth for 6 years and then was returned via the Space Shuttle in January 1990.
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Experiment within the Long Duration Exposure Facility (LDEF) (a STS Deployed Satellite)
Processing Facility: Solute Diffusion Apparatus: "...specially designed [chemical] reactors with three or more compartments separated by valves to keep the reactant solutions and solvent separated until apparatus reaches low gravity." (1, p.9)
Builder of Processing Facility: Rockwell International Science Center, Thousand Oaks, California

Experiment:
Growth of Crystals From Solutions in Low Gravity (A0139A)

The Long Duration Exposure Facility was a free-flying cylindrical structure (30 ft. long and 14 ft. in diameter) placed in orbit by the U.S. space shuttle at an altitude of 257 nautical miles and an inclination of 28.5°. The structure contained 57 science and technology experiments located in trays mounted on the exterior of the structure. LDEF was to be retrieved after approximately 9 months. However, the structure remained in orbit for nearly 6 years because shuttle flights were delayed following the loss of the space shuttle Challenger. LDEF was eventually retrieved at an altitude of approximately 180 nautical miles.

This Long Duration Exposure Facility (LDEF) experiment was the third in a series of investigations designed by Lind and/or Nielsen et al. to study the solution growth of organic crystals under low-gravity conditions (see Lind, ASTP; Nielsen, Spacelab 1 (this chapter)). The specific objective of the experiment was to produce high quality crystals using (1) the Opposite Oriented Diffusion (OOD) method and (2) the low-gravity environment provided by the LDEF.

Four specially designed reactors were employed for the experiments. Two of the reactors were constructed from stainless steel, the others from Teflon™. Each reactor contained three

chambers. Two of the chambers were reservoirs which contained the reactant solutions. Between the two reservoir chambers was a reactant chamber which contained a solvent. The solutions in each of the three chambers were separated by valves to prevent contact between the reactants prior to LDEF deployment. (The motorized valves were designed to prevent unwanted convection when opened.) See Reference (2) or (8) for a detailed discussion of the experimental apparatus.

The crystal growth experiments involved a solute diffusion process in which liquid reactant solutions diffused into pure solvent (in the reaction chamber) and reacted chemically to form single crystals. Three different growth systems were investigated during the LDEF Mission: (1) $CaCO_3$ (calcite-type) single crystals were grown from solutions of $CaCl_2$ and NH_4HCO_3 in water, (2) tetrathiofulvalene-tetracyanoquinidimethane (TTF-TCNQ) single crystals were grown from solutions of TTF and TCNQ in acetonitrile, and (3) PbS crystals were grown from $PbCl_2$ and CH_3CSNH_2 in water. (The $CaCO_3$ and PbS crystals were grown in the stainless steel reactors and the TTF-TCNQ experiments were grown in the Teflon™ reactors.)

These crystal growth experiments were performed on the LDEF because they require more low-gravity growth time than the 5-10 days available during a space shuttle flight. The experiment had been designed to operate for approximately 2 months, and then remain dormant for the remainder of the flight. Although the LDEF remained in orbit much longer than originally planned, the additional time did not have any significant effect on the crystal growth process or the crystals produced.

Reportedly, "The $CaCO_3$ experiment produced numerous clear, well-formed single or twinned crystals ranging between 1 and 6 mm in longest dimension, which is substantially larger than previous solution growth results. These crystals have an acute scalenohedral habit very different from the habits that we have obtained previously [on Earth and in space]. Their habit is like that of some natural calcite crystals, but very unlike the familiar rhombohedral habit of Iceland spar. The twinning is like that of natural calcite.

"The TTF-TCNQ experiment also produced numerous well formed single crystals. They are smaller than the largest crystals grown previously, but are adequate in size and quality for analysis. Their habit is also very different from that obtained previously [on Earth and in space]. The crystals are thin rectangular plates with the crystallographic b axis perpendicular to the plate. That is contrary to all our previous crystal growth experiments, in which the b axis has been the preferred direction of growth." (Reference (5)) <Note: Previous related space ex-

periments employing $CaCO_3$ and TTF-TCNQ are detailed in Reference (8).>

<Note: References (6), (7), (8) and (9) were received just prior to the publication of this technical memorandum. Thus, the documents were not available to aid in the preparation of this version of the experiment summary. However, additional information concerning the experiment as a whole can be found in these References.>

<Note: It was reported that results from the PbS crystal growth experiments were not as dramatic as those of the other two growth systems (Reference (10)). Results from these experiments did not appear to be detailed in any of the references listed below (including References (6)-(9)).>

Key Words: Crystal Growth From Solution, Reactant Solutions, Aqueous Solutions, Organic Crystals, Binary Systems, Ternary Systems, Insoluble Crystals, Single Crystals, Solvent, Diffusion, Double Diffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Liquid/Liquid Interface, Solid/Liquid Interface, Crystal Morphology, Crystalline Structure, Twinning, Platelet Habit, Free-Flying Satellite, Liquid Reservoir, Contained Fluids, Optics Applications, Semiconductor Applications, Semiconductors, Electronic Materials

Number of Samples: four
Sample Materials: (1) calcium carbonate, $CaCO_3$ grown from $CaCl_2$ and NH_4HCO_3 in water; (2) tetrathiofulvalene-Tetracyanoquinodimethane (TTF-TCNQ) grown from TTF and TCNQ in acetonitrile; (3) PbS grown from $PbCl_2$ and CH_3CSNH_2 in water.
(Ca*C*O*, Ca*Cl*, N*H*H*C*O*, Pb*S*, Pb*Cl*, C*H*C*S*N*H*)
Container Materials: stainless steel (two reactors) and Teflon™ (two reactors)

Experiment/Material Applications:
The three materials investigated for this study are important for research and technology applications: (1) PbS can be used as a semiconductor material, (2) $CaCO_3$ has useful optical properties, and (3) TTF-TCNQ is important because of its one-dimensional electrical conductivity.

See also Lind, ASTP.

References/Applicable Publications:

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(2) Lind, M. D. and Nielsen, K. F.: LDEF Experiments- Crystal Growth by a Solute Diffusion Process. In Proceedings of the 4th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 167-174. (preflight)

(3) Nielsen, K. F. and Lind, M. D.: Solution Crystal Growth on the Apollo-Soyuz, the Spacelab, the LDEF, and the Eureca Mission. Proceedings of the Fifteenth International Symposium on Space Technology and Science, Tokyo, 1986, pp. 2111-2116. (pre-recovery of LDEF samples)

(4) Solute Diffusion Apparatus. In Microgravity Science and Applications Experimental Apparatus and Facilities, developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, p. 32. (processing facility)

(5) Input received from Principal Investigator M. D. Lind, August 1993.

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(7) Lind, M. D. and Nielsen, K. F.: Crystal Growth by Solute Diffusion in Earth-Orbit. Proc. Crystal Growth in Space and Related Optical Diagnostics, SPIE-The International Society for Optical Engineering, July 22-23, 1991, San Diego, California, Vol. 1557, pp. 259-270. (post-flight)

(8) Nielsen, K. F. and Lind, M. D.: Results of the TTF-TCNQ and the Calcium Carbonate Crystallization on the Long Duration Exposure Facility. Proc. LDEF- 69 Months in Space, First Post-Retrieval Symposium, Kissimmee, Florida, June 2-8, 1991, NASA CP-3134, pp. 1675-1683. (post-flight)

(9) Nielsen, K. F., Lind, M. D., Gerward, L., and Thorup, N.: Crystal Growth of TTF-TCNQ by Solute Diffusion in Earth-Orbit. Physica Scripta, Vol. 47, pp. 596-598, 1993. (post-flight)

(10) Personal communication with M. D. Lind, September 1993.

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Co-Investigator(s): Iwai, S. (2)
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Experiment Origin: Japan
Mission: TT-500A 10 (Materials Processing Flight #3)
Launch Date/Expt Date: August 1981
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: One electric furnace
Builder of Processing Facility: Ishikawajima Harima Co.-Ltd. <Note: Location unknown>

Experiment:
Pb-Sn-Te Single Crystal Growth

This TT-500A experiment was the first in a series of investigations designed by Segawa et al. to study the low-gravity solidification of a Pb-Sn-Te semiconductor.

It appears that the experimental objectives and setup could were similar to those outlined under Segawa's later Pb-Sn-Te experiment (see Segawa, TT-500A 12).

Reportedly, although the rocket was successfully launched, "...the TT-500A rocket [was] not recovered because...a main parachute...[was]...not activated...." (1, p. 2)

No further details appear to be available concerning (1) the reported parachute anomaly or (2) the experimental results. <Note: It was not clear if this rocket was equipped to provide real-time data to principal investigators on the ground during the reduced gravity flight. If such real-time data were available, some experimental results might have been observed by the investigator.>

<Note: Although it appeared that this experiment could be included in the "Crystal Growth From the Melt" chapter (Chapter 9), Segawa indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Melt and Solidification, Ternary Systems, Single Crystals, Substrates, Growth Kinetics, Solid/Liquid Interface, Crystal Morphology, Surface Morphology, Crystalline Structure, Semiconductors, Semiconductor Applications, Electronic Materials, Payload Survivability, Payload Recovery System Failure

Number of Samples: one
Sample Materials: lead-tin-telluride (same as TT-500A 12)
(Pb*Sn*Te*)
Container Materials: quartz (evacuated crucible)
(Si*O*)

Experiment/Material Applications:
See Segawa, TT-500A 12.

References/Applicable Publications:
(1) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. International Astronautical Federation, 34th International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper 83-157, 9 pp. (very short summary; post-flight)

(2) Input received from Principal Investigator Y. Segawa, August 1993.

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Experiment Origin: Japan
Mission: TT-500A 12 (Materials Processing Flight #5)
Launch Date/Expt Date: January 1983
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: One electric furnace
Builder of Processing Facility: Ishikawajima Harima Co.-Ltd. <Note: Location unknown)

Experiment:
Pb-Sn-Te Single Crystal

This TT-500A experiment was the second in a series of investigations designed by Segawa et al. to study the low-gravity solidification of a PbSnTe semiconductor (see Segawa, TT-500A 10). The specific objectives of the experiment were to obtain (1) a single Pb-Sn-Te crystal of higher quality and larger size than can be produced on Earth and (2) a better understanding of the kinetics of crystal growth. To achieve these objectives, two types of crystal growth were attempted: (1) epitaxial growth of PbSnTe on a PbTe substrate and (2) homogeneous nucleation and growth of PbSnTe at the free surface of a Pb solution.

<Note: The following description of the sample material was somewhat unclear to the editors.> Prior to the rocket flight, the experiment sample material was prepared. "The substrate for the epitaxial growth was a (100) surface of the PbTe single crystal. The... [substrates] were cut from the ingot, polished, and etched by HBr + Br_2(5%) etchant for 1 minute. The Pb melt (Pb 0.5 g, Sn 0.075 g, Te 0.15 g) was fused at 620 C in purified H_2 gas for 5 hours. This melt was kept in contact with the substrate at 600 C for 10 minutes. After cooling, the substrate and the Pb melt was [sic] enclosed in vacuum by a... [quartz] capsule." (1, p. 294)

During the rocket flight, one electric furnace was used to process the sample. Approximately 80 seconds after rocket launch the furnace was switched on and the material was heated to 650 ^{o}C (50 ^{o}C over the intended temperature). After rapid cooling to 600 ^{o}C, the material was held at this point for about 4 minutes. Cooling below the Pb melt temperature was then accomplished by application of He gas.

Post-flight examination of the material revealed that the surface of the Pb melt was round, indicating solidification under low-gravity conditions. While sometimes, when Pb is melted on Earth, the Pb will react with the quartz and the transparent ampoule surface becomes opaque; this did not occur in the low-gravity sample, which suggested that the sample solidified without contacting the quartz wall.

Metallographic examination of the PbTe substrate indicated a meltback caused by the temperature overshoot. Squares of epitaxially grown $Pb_{0.7}Sn_{0.3}Te$ were found (Sn composition determined by x-ray microanalysis). Examination of the Pb melt revealed squares of PbSnTe crystals covering the surface.

Surface tension forces resulted in a slightly convex PbSnTe surface. "Also we can see some parallel faces... suggesting that these crystals have the same crystalline axis. [In] Other words, they are the [sic] thin single crystal in spite of... some cracks caused by the convex curve of the surface." (1, p. 296) X-ray Laue patterns indicated the PbSnTe thin film had a (100) surface. Other crystalline axes also had the same orientation. This characteristic was not observed in 1-g processed materials.

Reportedly, without convection, under low-gravity conditions, nuclei were not moved throughout the melt. Therefore, a thin film grew on the melt surface. Eventually, this film covered the entire surface. This suggested that low-gravity conditions will permit the formation of a thin, single crystalline film.

<Note: Although it appeared that this experiment could be included in the "Crystal Growth From the Melt" chapter (Chapter 10), Segawa indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Melt and Solidification, Metallic Solutions, Ternary Systems, Single Crystals, Substrates, Epitaxial Growth, Free Surface, Wetting, Surface Tension, Meniscus Shape, Growth Kinetics, Buoyancy Effects Diminished, Homogeneous Nucleation, Nucleation, Solid/Liquid Interface, Quench Process, Crystal Morphology, Surface Morphology, Crystalline Structure, Cracks, Films, Thin Films, Film Microstructure, Sample Detachment From Crucible, Coated Surfaces, Non-Wetting of Container, Thermal Environment More Extreme Than Predicted, Furnace Malfunction, Semiconductors, Semiconductor Applications, Electronic Materials

Number of Samples: one
Sample Materials: lead-tin telluride
(Pb*Sn*Te*)
Container Materials: quartz (evacuated crucible)
(Si*O*)

Experiment/Material Applications:
PbSnTe is mixed crystal of PbTe and SnTe. The band gap energy of $Pb_{1-x}Sn_xTe$ approaches 0 eV near the mole ratio of x = 0.3. Thus, the material is of interest for opto-electronic applications in the infrared region.

References/Applicable Publications:
(1) Segawa, Y., Iwai, S., Namba, S., Kajiwara, K., Akimoto, T., Masuda, T., Shibatou, Y., and Matuda, T.: Crystal Growth of PbSnTe Under Microgravity. In Second Joint Japan-Germany-ESA Symposium on Microgravity Research, Tokyo, March 25-26, 1985, Summary Report, pp. 293-306. (post-flight)

(2) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. International Astronautical Federation, 34th International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary; post-flight)

(3) Input received from Principal Investigator Y. Segawa, August 1993.

(4) Segawa, Y.: Oral Presentation, 1983 Autumn Meeting of the Applied Physics Society of Japan, 28a-E-10. (in Japanese)

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Experiment Origin: USA
Mission: STS Launch #5, STS-005 (STS 31-A, Columbia)
Launch Date/Expt. Date: November 1982
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment, Shuttle Student Involvement Program (SSIP), Middeck Experiment (Stowage Locker)
Processing Facility: Hot and cold plate charged with saturated solution
Builder of Processing Facility: Hamilton Standard Division of United Technologies, Windsor Locks, Connecticut

Experiment:
Crystal Growth of Triglycine Sulfate (SE81-5)

During the terrestrial processing of triglycine sulfate (TGS), gravity-induced forces contribute to crystalline defects in the solidifying material.

This STS-005 Shuttle Student Involvement Program Experiment was designed to study TGS crystal growth under low-gravity conditions. The specific objectives of the research were to (1) compare Earth-grown and space-grown TGS crystals, and (2) determine if perfect TGS crystals could be produced in space.

<Note: The description of the processing facility (as provided in References (1) and (2)) was somewhat unclear:> The self-contained, battery-powered experiment apparatus was configured in a shuttle middeck locker. The apparatus consisted of a hot plate (solute cup) and a cold plate (sting) maintained by small thermoelectric heat pumps. Heat was extracted from the cold sting upon which a seed crystal was glued. "Imbedded in the solute cup was a cavity containing solute enrichment crystals, which were separated from the growth chamber by a screen." (2, p. 3) Prior to the shuttle launch, "the cell" was filled with TGS solution.

During the first hours of the shuttle mission, the experiment was initiated by a switch. As planned, the apparatus operated until battery drain terminated the experiment. After landing and apparatus disassembly, the crystalline contents were sent to Hamil-

ton Standard and NASA Lewis Research Center for tests and analysis.

Post-flight analysis of the payload indicated that "Rather than a single perfect crystal, a crystal cluster formed in an upper corner of the fixture. This made it impossible to analyze the crystals for size, clarity, shape, etc. as the mass had to be broken apart to be removed....

"Analysis of the final sample suggests that Shuttle temperatures may have exceeded experiment constraints as the results were not as expected. Unexpected bacterial contamination during landing also softened the seed crystal. A poor vacuum and lack of solute convection probably contributed to the failure as well." (2, p. 3)

Key Words: Crystal Growth From Solution, Saturated Solution, Seed Crystals, Single Crystals, Cooled Sting Technique, Density Difference, Thermal Gradient, Solutal Gradients, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Heat and Mass Transfer, Thermosolutal Convection, Buoyancy-Driven Convection, Absence of Buoyancy Forces (Detrimental), Solid/Liquid Interface, Surface Morphology, Crystal Morphology, Crystalline Defects, Contamination Source, Thermal Environment More Extreme Than Predicted, Contained Fluids, Vacuum, Infrared Detector Applications, Optics Applications, Electronic Materials

Number of Samples: unknown, probably one
Sample Materials: saturated solution of triglycine sulfate (TGS) (N*H*C*H*C*O*H*, H*S*O*)
Container Materials: unknown

Experiment/Material Applications:
"Since tri-glycine sulfate crystals are used extensively in sensors and imaging devices, improving their quality could have significant benefit.... And a "perfect" tri-glycine sulfate crystal would yield a much purer light from a laser beam. TGS crystals are also used to detect infrared rays radiating from the ground during satellite observations of the Earth's surface." (2, p. 1)

See also Lal, STS-024.

References/Applicable Publications:

(1) STS-5 Fifth Space Shuttle Mission. NASA Press Kit, October 1982, p. 40. (preflight)

(2) The Formation of Crystals in a Weightless Environment. In Shuttle Student Involvement Program (SSIP) Final Reports of Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) Canister G-002
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-002: German Youth Fair/Kayser-Threde, Munich, Germany
Processing Facility: Thermally isolated experiment fluid cell with four foil heaters; interferometer
Builder of Processing Facility: Kayser-Threde GmbH, Munich, Germany

Experiment:
Crystal Growth in Liquid Salt Solution

This experiment was one of five investigations housed within the G-002 Get Away Special Canister on STS-007. (One other experiment (of the five) was applicable to this data base (see Riepl, STS-007 (Chapter 15)).) The specific objective of the experiment was to grow crystals from a potassium phosphate salt solution in a reduced gravity environment.

The investigation was performed in a single, thermally isolated experiment cell with four orthogonal optical windows. Reportedly, the cell was capable of containing approximately "160 cubic centimeters" of liquid KH_2PO_4 salt solution. <Note: It appears this solution also contained salt crystals.> The cell was configured with (1) four, 20 W foil heaters, (2) a fluid mixer, (3) a neoprene membrane, and (4) a nylon rope (which extended across the center of the cell and acted as an initiator for crystal growth). <Note: The purpose of the neoprene membrane was not specified.> The cell was "...integrated into a laser Doppler interferometer with an orthogonally splitted [sic] He-Ne laser beam of approx. 0.5 mW output power." (2, p. 137)

A detailed optics arrangement in the payload allowed a single camera to document interferometric results (see 2, p. 137). (Interferograms are used to evaluate the solutal density distribution about the growing crystal.)

A figure in Reference (2) illustrated (in part) the experiment sequence. During the first hour of the experiment, salt crystals began to dissolve as the cell was heated from approximately 15 ^{o}C to 55 ^{o}C. During the following 1.8 hours, (1) the dissolving process continued, (2) the solution was mixed with the fluid mixer, and (3) the cell was heated from 55 ^{o}C to approximately 82 ^{o}C. At the end of this 1.8 hours, the resultant solution was allowed to freely cool for 0.75 hour (to a temperature of approx. 65 ^{o}C). During the following experimental sequence, a controlled cooling (temperature gradient) period of the cell was realized: (1) the solution temperature dropped from 65 ^{o}C to its saturation temperature of 60 ^{o}C (within about 0.5 hour), and (2) continued to steadily drop until the (nearly) ambient payload temperature had been attained (about 6 hours).

During the controlled cooling period (and possibly before this period), two interferograms of the experiment cell were obtained every 4 minutes. These two interferograms were recorded, one right after another, on the single camera (with a time delay of 10 seconds between photos). The two interferograms were taken from orthogonal views (a total of 250 interferograms were taken by the available camera).

Post-flight analysis indicated that "...the experiment performed as expected, the predetermined temperature profile was achieved, a crystal was growing in the center of the cell, and stills of interferograms were obtained." (2, p. 138) It was reported that one optical window was partially obscured by salt crystals which did not dissolve during the preheating phase.

<Note: Reference (1) reported that "...some unexpected gas bubbles caused disturbancies [sic] in the density structures," while Reference (6) reported that "...some unexpected gas bubbles caused density variations in the salt crystal." Because no more detailed information was available, it is likely that gas bubbles were present in the solution and these bubbles affected the density distribution about the growing crystal.>

No further information on the evaluation of the grown crystal could be located at this time.

Key Words: Crystal Growth From Solution, Salt Crystals, Salt Solution, Solutal Gradients, Thermal Gradient, Density Distribution, Thermosolutal Convection, Diffusion, Mass Transfer, Stirring of Components, Liquid Mixing, Saturation, Bubbles, Bubble Formation, Passive Cooling, Solid/Liquid Interface, Crystalline Structure, Crystal Morphology, Coated Surfaces, Contained Fluids, Interferometric Measurement

Number of Samples: one
Sample Materials: KH_2PO_4 (potassium-dihydrogen-phosphate) (160 cc liquid salt solution)
(K*H*P*O*)
Container Materials: Aluminum (the inner surface was Teflon™-coated).
(Al*)

Experiment/Material Applications:
This research was designed to illustrate the solutal density structure surrounding a growing crystal in the reduced gravity environment.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS-7, JSC-17645 Annex STS-7, April 1, 1983. (short description; preflight)

(2) Schmitt, G.: The G-002 JUFO-1 Payload, Its Objectives and Results. In NASA Goddard Space Flight Center's 1984 Getaway Special Experimenter's Symposium, August 1-2, 1984, pp. 135-142, NASA CP-2334. (post-flight)

(3) STS-7 Seventh Space Shuttle Mission. NASA Press Kit, June 1983, p. 55. (short description; preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

(5) Input received from K. Kemmerle, December 1989.

(6) "STS-7 Getaway Specials." NASA News, NASA/GSFC, May 1983.

(7) Input received from G. Schmitt (Kayser-Threde), July 1993.

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Experiment Origin: France
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1, Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility
Processing Facility: Thermostatically controlled facility containing four, three-chambered chemical reactors. The facility was shared with Spacelab Experiment 1ES332.
Builder of Processing Facility: Builder of thermostat: B W Electronics, Aarhus, Denmark; builder of cartridges: COMAT-Balma, France

Experiment:
Crystal Growth by Co-Precipitation in Liquid Phase 1ES333
Brushite: Experiments 1 & 2, Lead Phosphate: Experiments 3 & 4

On Earth, both convection and diffusion drive fluid mass transfer during low temperature solution crystal growth. Because "...convective currents are known to produce saturation jumps which can generate growth defects..." (1, p. 193), this Spacelab experiment sought to alleviate gravity-driven convection effects and thus achieve diffusion-dominated crystal growth.

During the mission, four, three-compartment cartridges, held in a thermostatically controlled facility, were employed to grow insoluble crystals of brushite (calcium nitrate + hydrogen ammonium phosphate) and lead monetite (lead nitrate + phosphoric acid) using a double diffusion technique. (Two, three-compartment cartridges were dedicated to the brushite system, two, three-compartment cartridges to the lead monetite system.) During the investigation, reactants held in the two outer compartments of each cartridge were allowed to diffuse toward the third, central compartment using a valving system. The resulting crystals were formed in the central compartment.

Simulation of the space experiment in the ground-based laboratory was completed for comparison. During this terrestrial experiment, gravity-driven convective effects were reduced by growing the crystals in a three-layer configuration employing a gel medium.

It appears that both the 1-g and low-g crystals were characterized by optical and x-ray topographic techniques. Reportedly, brushite and lead monetite have a "...high growth rate which develops a platelet habit; this habit is particularly convenient for X-ray topographic characterization... of defects." (1, p. 193).

Platelet habit was observed in space crystals grown in both calcium phosphate cartridges and in one lead phosphate cartridge. In the fourth cartridge (lead phosphate), crystals were not platelets, rather "...most of these crystals were heterogeneously nucleated on the walls of the cartridge nearby the nitrate compartment." (1, p. 197)

After characterization of the space grown crystals, it was concluded that convenient diffusive regimes had been obtained in gel-free media in the reduced gravity environment of Spacelab. Dielectric measurements on lead hydrogen phosphate crystals illustrated an improvement of crystalline quality of space-grown crystals when compared to similar gel-grown crystals.

Many other observations were presented in Reference (1).

Key Words: Crystal Growth From Solution, Reactant Solutions, Insoluble Crystals, Model Materials, Diffusion, Double Diffusion, Diffusion-Controlled Growth, Diffusive Mass Transfer, Liquid/Liquid Diffusion, Mass Transfer, Buoyancy-Driven Convection, Precipitation, Saturation, Growth Rate, Solid/Liquid Interface, Crystalline Defects, Surface Morphology, Platelet Habit, Nucleation, Heterogeneous Nucleation, Nucleation Sites, Crucible Effects, Material Interaction with Containment Facility, Liquid Reservoir, Contained Fluids

Number of Samples: four
Sample Materials: case of brushite: [$Ca((NO_3)_2)$ (calcium nitrate) + $NH_4H_2PO_4$ (hydrogen ammonium phosphate)]; case of lead monetite: $Pb(NO_3)_2$ (lead nitrate) + H_3PO_4 (phosphoric acid); ground-based gel media: tetramehoxysilane (TMS)
(Ca*N*O*N*H*P*O*, Pb*N*O*H*P*O*)
Container Materials: cartridges: Lexan™

Experiment/Material Applications:
Originally, a manganese carbonate system, rather than a lead phosphate system, was to be examined. When time allocated for the flight experiment was restricted, the phosphate system was substituted. The phosphate crystals have a significantly higher growth rate which allowed crystals of reasonable size to be grown in the time allowed.

The chosen crystals are model materials suitable to test the process of double-diffusive solution crystal growth in a low-gravity environment. "Insoluble crystals of compounds with complicated chemical composition include several materials with interesting properties. Most of these compounds decompose at lower temperatures so that melt or vapour growth techniques cannot be applied. Classical solution growth does not apply either, because these compounds are at the best only slightly soluble. Precipitation by interdiffusion of two solutions appears theoretically the only remaining possibility." (4, p. 440)

References/Applicable Publications:
(1) Robert, M. C., Lefaucheux, F., and Authier, A.: Growth and Characterization of Brushite and Lead Monetite Simulation and Results. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 193-199. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering publication (acceleration measurements on Spacelab 1)

(3) Robert, M. C., Lefaucheux, F., Jannot, B., Godefroy, G., and Garnier, E.: A Comparative Study of Gel Grown and Space Grown Lead Hydrogen Phosphate Crystals. Journal of Crystal Growth, 88 (1988), pp. 499-510. (post-flight)

(4) Kaldis, E.: Crystal Growth from Solutions and Nucleation from the Vapour Phase. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 439-441. (post-flight)

(5) Presenti, P.: Orientation and Perspectives of the French Materials Science in Space Programme. International Astronautical Federation, 30th International Astronautical Congress, Munich, West Germany, September 17-22, 1979. (preflight; manganese carbonate system discussed)

(6) Input received from Principal Investigator M. C. Robert, September 1988.

(7) Input received from Experiment Investigator, June 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: Radiation from two halogen lamps was focused on a sample by two half ellipsoidal mirrors.
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Growth of Semiconductor Crystals by the Travelling Heater Method (GaSb) (1ES323)

When binary III-V semiconductors are grown on Earth, buoyancy-driven convection in the melt results in intensive dopant inhomogeneities.

This experiment was the first in a series of investigations designed by Benz et al. to study the growth of semiconductor crystals using the Travelling Heater Method (THM). The specific objective of the experiment was to determine if Te-doped GaSb THM-grown crystals exhibited a reduction in (gravity-induced convective) dopant inhomogeneities.

In the THM method employed here, growth from a metallic solution is realized rather than growth from a melt. Among other advantages, the THM lower growth temperature reduces vapor pressure difficulties as well as reduces contamination of the sample from the ampoule.

During the mission, a solution zone of gallium with 3% Sb was placed between a solid GaSb seed (111) and solid GaSb polycrystalline feed material and heated in the Mirror Heating Facility (MHF) furnace. Reportedly, "By moving the mirror heating furnace with respect to the fixed sample, crystallization of the advancing seed/solution interface can be achieved. Simultaneously feed material is dissolved at the feed/solution interface. The solute transport in the liquid zone may be established by diffusion and/or convection." (1, p. 158)

Reportedly, because only 9.5 hours of the planned 24 hours growth time was realized, a smaller than anticipated, 220 micron sample was produced. When simulation of the Spacelab 1 experiments was completed on Earth, a similarly sized crystal was produced.

In comparison to the Earth-processed sample, the space-grown crystal had reduced dopant inhomogeneities; only a few rotational striations were observed in the first 40 microns of the sample. In the middle section of the space crystal, nonrotational striations occurred only in the vicinity of the crystal imperfections. (Such striations were distributed over the entire cross section of the Earth processed sample.) The space crystal was nearly striation free in the last section of the sample.

<Note: Although it appeared that this experiment could also be included in the "Crystal Growth From the Melt" chapter, Reference (6) indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Metallic Solutions, Solution Zone, Binary Systems, Ternary Systems, Model Materials, Dopant, Dopant Distribution, Seed Crystals, Feed Material, Single Crystals, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Solid/Liquid Interface, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Striations, Rotational Striations, Semiconductors, Semiconductor Applications, Electronic Materials, Processing Time Not as Long As Planned, Halogen Lamps

Number of Samples: one
Sample Materials: tellurium doped GaSb
seed material: GaSb (111)
feed material: GaSb polycrystalline
(Te*Ga*Sb*)
Container Materials: quartz ampoules (fused silica)
(Si*O*)

Experiment/Material Applications:
GaSb is a model system for crystal growth of III-V semiconductors. The aim of the research was to improve the crystal quality. Inhomogeneities and convective transport phenomena under Earth and reduced-gravity conditions were investigated.

Direct applications of the semiconductors include optoelectric devices such as light emitting diodes, lasers, field effect transistors, etc. For all applications, crystals with a well-defined electrical behavior are desired.

References/Applicable Publications:

(1) Benz, K. W. and Nagel, G.: GaSb Semiconductor Crystals Under Microgravity, Experiment ES 323. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 157-161. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48, Teledyne Brown Engineering publication. (acceleration measurements on Spacelab 1)

(3) Nagel, G. and Benz, K. W.: Travelling Heater Growth of GaSb Under Reduced Gravity During the First Spacelab Mission. Advances in Space Research, Vol. 4, No. 5, pp. 23-26, 1984. (post-flight)

(4) Eyer, A., Nitsche, R., and Zimmermann, H.: A Double-Ellipsoidal Mirror Furnace for Zone Crystallization Experiments in Spacelab. Journal of Crystal Growth, 47 (1979), pp. 219-229. (furnace setup)

(5) Benz, K. W., Danilewsky, A., Notheisen, B., and Nagel, G,: Growth of III-V Semiconductors by the Travelling-Heater-Method Under Microgravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission, D1, Norderney, Germany, August 27-29, 1986, pp. 275-280. (discusses Spacelab 1 results as well)

(6) Input received from A. N. Danilewsky (Kristallographisches Institut der Universität, Freiburg), July 1989 and August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: Radiation from two halogen lamps was focused on the sample by two half ellipsoidal mirrors. (This furnace was also used on Spacelab 1.)
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Travelling Heater Method (Te doped GaSb) - Growth of Single Crystals of Binary III-V Semiconductors (WL-MHF 02)

This experiment was the second in a series of investigations designed by Benz et al. to study the growth of semiconductor crystals using the Traveling Heater Method (THM) (see Benz, Spacelab 1). The specific objective of the experiment was to determine if Te doped GaSb THM-grown crystals exhibited a reduction in (gravity-induced convective) dopant inhomogeneities. Reportedly, the investigation was very similar to the earlier THM experiment by Benz et al. flown on Spacelab 1.

During the Spacelab D1 experiment, growth from a metallic solution was realized in the Spacelab Mirror Heating Facility (MHF). The entire (planned) sample growth time of 24 hours was achieved, resulting in a crystal 300 microns long. The space-processed sample was compared to similarly processed ground-based reference samples.

Reportedly, the space-grown Te doped GaSb crystal was found to be nearly striation free with only residual dopant inhomogeneities, while Earth-processed crystals showed "...pronounced structures of rotational and non-rotational periodic striations over the whole cross section of the crystal...." (3, p. 278) It was concluded that "Metallic solution growth in space, therefore, offers the possibility of comparing kinetic-related growth properties with effects which are influenced by the growth solution." (3,

p. 280)

<Note: Although it appeared that this experiment could also be included in the "Crystal Growth From the Melt" chapter, Reference (6) indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Metallic Solutions, Solution Zone, Ternary Systems, Model Materials, Dopant, Dopant Distribution, Seed Crystals, Feed Material, Single Crystals, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Growth Kinetics, Solid/Liquid Interface, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Striations, Semiconductors, Semiconductor Applications, Electronic Materials, Halogen Lamps

Number of Samples: one
Sample Materials: GaSb doped with Te
(Ga*Sb*Te*)
Container Materials: closed quartz glass ampoule
(Si*O*)

Experiment/Material Applications:
See Benz, Spacelab 1

References/Applicable Publications:
(1) Benz, K. W., Danilewsky, A., and Nagel, G.: Growth of III-V-Semiconductors By the Travelling Heater Method Under Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, pp. 85-86. (abstract only)

(2) Benz, K. W.: III-V Compound Semiconductor Growth by the Travelling Heater Method (THM) Under Reduced Gravity. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 115-116. (preflight)

(3) Benz, K. W., Danilewsky, A., Notheisen, B., and Nagel, G.: Growth of III-V Semiconductors by the Travelling Heater Method Under Microgravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 275-280. (post-flight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight, acceleration measurements on D1)

(5) Benz, K. W., Danilewsky, A., Notheisen, B., and Nagel, G.: Growth of III-V Semiconductors by the Travelling Heater Method Under Microgravity. In Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, (1987), pp. 345-347. (D1 and Spacelab results)

(6) Input received from A. N. Danilewsky (Kristallographisches Institut der Universität), July 1989 and August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, MEDEA Double Rack
Processing Facility: Monoellipsoidal Mirror Furnace (ELLI)
Builder of Processing Facility: Dornier-System GmbH, Germany

Experiment:
Travelling Heater Method (S doped InP) - Growth of Single Crystals of Binary III-V Semiconductors (MD-ELI 01)

This Spacelab D1 experiment was the third in a series of experiments designed by Benz et al. to study the growth of semiconductor crystals using the Travelling Heater Method (THM) (see Benz, Spacelab 1 and Spacelab D1 ("Travelling Heater Method Te doped GaSb")). The specific objective of the experiment was to determine if S-doped InP THM-grown crystals exhibited a reduction in (gravity-induced convective) dopant inhomogeneities.

InP crystals were to be grown during the mission at a temperature of 850 ^{o}C. However, a malfunction of the ELLI furnace prevented the exact temperature of the zone to be determined. A crystal 3.7 mm in length was produced.

Sample analysis indicated that "In the middle of the crystal a bubble has been generated, which led to a polycrystalline growth regime... parts of the grown crystal are single crystalline with a few small In-inclusions." (3, p. 277)

Reportedly, the experiment demonstrated that the S-doped crystals could be grown nearly striation free. "Residual dopant inhomogeneities in the space grown [crystal]... which were due to rotational effects, growth kinetics (type II striations) etc. are less intensive as compared to 1-g reference experiments. Metallic solution growth in space, therefore, offers the possibility of comparing kinetic-related growth properties with effects which are influenced by the growth solution." (3, p. 280)

<Note: Not all the publications listed below were available to the authors of this document at the time this experiment summary was prepared.>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Metallic Solutions, Solution Zone, Ternary Systems, Dopant, Dopant Distribution, Seed Crystals, Feed Material, Single Crystals, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Growth Kinetics, Solid/Liquid Interface, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Bubbles, Bubble Formation, Striations, Rotational Striations, Inclusions, Semiconductors, Semiconductor Applications, Electronic Materials, Furnace Malfunction

Number of Samples: one
Sample Materials: indium-phosphide doped with sulfur
seed material: sulfur-doped InP (111)
feed material: sulfur-doped InP
(In*P*S*)
Container Materials: quartz glass ampoule
(Si*O*)

Experiment/Material Applications:
See Benz, Spacelab 1.

References/Applicable Publications:
(1) Benz, K. W., Danilewsky, A., and Nagel, G.: Growth of III-V Semiconductors by the Travelling Heater Method Under Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, pp. 85-86. (post-flight; abstract only)

(2) Benz, K. W.: III-V Compound Semiconductor Growth by Travelling Heater Method (THM) Under Reduced Gravity. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 115-116. (preflight)

(3) Benz, K. W., Danilewsky, A., Notheisen, B., and Nagel, G.: Growth of III-V Semiconductors by the Travelling-Heater Method Under Microgravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 275-280. (post-flight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements on D1)

(5) Authier, A., Benz, K. W., Robert, M. C., and Wallrafen, F.: Fluid Sciences and Materials in Space. Edited by H. U. Walter, Springer Verlag, 1987, p. 405.

(6) Benz, K. W.: German Crystal Growth Experiments in Space. Jap. J. Appl. Phys., Vol. 57, No. 10 (1988).

(7) Danilewsky, A. N., and Benz, K. W.: InP-Growth from In Solutions Under Reduced Gravity. J. Crystal Growth, 97 (1989), pp. 571-577.

(8) Input received from A. N. Danilewsky (Kristallographisches Institut der Universität), July 1989 and August 1993.

(9) Danilewsky, A. N., Benz, K. W., and Nishinaga, T.: Growth Kinetics in Space and Earth-Grown InP and GaSb Crystals. J. Crystal Growth, 99 (1990), pp. 1281-1286.

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Experiment Origin: Denmark
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack #11
Processing Facility: Thermostatically controlled chamber containing several three-chambered chemical reactors. The facility was shared with Experiment 1ES333.
Builder of Processing Facility: Unknown, possibly Technical University of Denmark, Lyngby, Denmark

Experiment:
Organic Crystal Growth 1ES332

Slightly soluble or even insoluble substances may be grown from solution by the Opposite Oriented Diffusion (OOD) method. The method allows diffusion of two or more reactant solutions through a solvent toward a central area where a crystal is precipitated. On Earth, both the transport process and crystal growth are heavily influenced by gravity-driven convection. This convection can cause the growth of (1) many small crystals and (2) crystals with an irregular array of dislocations. It was expected that a reduction in convective flow (made possible in a low-gravity environment) would allow the production of (1) larger crystals, (2) crystals with fewer dislocations, and (3) crystals containing regular dislocation patterns.

This Spacelab 1 experiment was the second in a series of investigations designed Lind and/or Nielsen et al. to study the solution growth of organic crystals under low-gravity conditions (see Lind, ASTP (this chapter)).

Initially, it was proposed that crystals of the type TTF-TCNQ (TetraThiaFulvalene-TetraCyNoQuinodimethane) should be processed. The TTF-TCNQ crystal is of interest to solid state physicists because of its unique single crystal conductivity properties. Because this crystal growth system required acetonitrile as a solvent, and Spacelab safety requirements did not permit the use of acetonitrile, growth systems using either water or weak acid as a solvent were eventually selected.

Prior to the Spacelab 1 flight, 12 reaction chambers were prepared to study three different crystal growth systems. Each reactor contained three chambers: two reservoir chambers and one reaction chamber. Each reservoir chamber contained a reactant solution. The chambers were configured with a rubber wall to suppress air bubbles and provide equal pressure for the three chambers.

During the Spacelab 1 mission, crystal growth of the following systems were studied: (1) a platinum complex, $(NH_4)_{1.6}Pt(C_2O_4)_2$; (2) calcium tartrate, $CaC_4H_4O_6$; (3) calcium carbonate, $CaCO_3$. The experiments were initiated by opening valves between the chambers in each of the reactors. After the valves were open, the appropriate reactant solutions diffused into the central reaction chamber and the two solutions combined to form the intended single crystal. <Note: Half of the reaction chambers used valves with teflon filters. On Earth, these teflon filters are used to prevent unwanted convection which may occur when the valves are opened.> The temperature of the reaction chambers was maintained at 40 ^{o}C.

Post-flight, it was reported that the experiment proceeded as planned (temperature control, opening of the valves). "However, during the prolonged waiting period prior to launch, the Platinum-complex molecules desintegrated [sic] chemically, and the low-dimensional crystal growth experiments did not yield any useful results. Four of the remaining six experiments were successful. Two of those grew Calcium-Carbonate, two grew Calcium-Tartrate." (1, p. 190)

Of the successful experiments, only the results from the calcium-tartrate growth system were reported. It appears that the employed teflon filters permitted a slower and more controlled growth rate of the calcium-tartrate crystals. "It is obvious from the results that filters were necessary between the reservoirs and the growth chamber.... This is indicative of some g-levels being present during the mission causing too much convection in the crystallization chamber with no filter protection." (1, p. 191) <Note: More detailed reporting of the experimental results could not be located.>

Key Words: Crystal Growth From Solution, Reactant Solutions, Aqueous Solutions, Organic Crystals, Insoluble Crystals, Single Crystals, Solvent, Diffusion, Double Diffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Precipitation, Liquid/Liquid Interface, Solid/Liquid Interface, Acceleration Effects, Crystal Morphology, Crystalline Structure, Dislocations, Bubbles, Liquid Reservoir, Contained Fluids, Deterioration of Loaded Samples Prior to Launch, Optics Applications, Electronic Materials, Electrical Conductivity

Number of Samples: twelve
Sample Materials: six $(NH_4)_{1,6}Pt(C_2O_4)_2$ (platinum complex); four $CaC_4H_4O_6$ (calcium-tartrate); two $CaCO_3$ (calcium carbonate) (N*H*Pt*C*O*, Ca*C*H*O*, Ca*C*O*)
Solvent Material: Water for platinum-complex crystals and sulfuric acid, 0.05 M H_2SO_4, for the two remaining crystal systems (H*S*O*)
Container Materials: rubber

Experiment/Material Applications:
The specific reason why the platinum complex was selected was not detailed in the available publications. However, the specific reasons why calcium-carbonate and calcium-tartrate were used can be found under Lind, ASTP (this chapter).

References/Applicable Publications:
(1) Galster, G. and Nielsen, K. F.: Crystal Growth From Solutions. In ESA 5th European Symposium On Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 189-191. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering publication (acceleration measurements on D1)

(3) Nielsen, K. F. and Lind, M. D.: Solution Crystal Growth on the Apollo-Soyuz, the Spacelab, the LDEF, and the EURECA Missions. Proceedings of the Fifteenth International Symposium on Space Technology and Science, Tokyo, 1986, pp. 1111-1116.

(4) Kaldis, E.: Crystal Growth from Solutions and Nucleation from the Vapour Phase. In Proceedings of the 5th European Symposium on Material Sciences under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 439-441. (post-flight)

(5) Nielsen, K. F.: Diffusion Growth of Organic Charge-Transfer Crystals. In Material Sciences in Space, ESA SP-114, 1976, pp. 255-258. (preflight)

(6) Nielsen, K. F.: Crystal Growth in the Microgravity Environment. In Symposium on Industrial Activity in Space, Stresa, Italy, May 2-4, 1984, pp. 306-320. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack
Processing Facility: Mirror Heating Facility (MHF) Furnace: Radiation from halogen lamps is focused on sample by two ellipsoidal mirrors
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Growth of Cadmium Telluride by Traveling Heater Method (1ES322)

The traveling heater method (THM) is a process in which a heated solution zone passes through a polycrystalline feed rod to grow a single crystal. Generally, the process involves heating the solution zone by radiation from halogen lamps located in an ellipsoidal mirror furnace. The THM ampoule, which contains the solution zone, feed rod, and crystal, is then translated through the common focus of the mirrors. This process allows substantial lowering of the crystal growth temperatures (compared to stoichiometric melt methods) and a corresponding reduction in crystal defect density. (See Reference (3) for further discussion of the processing method.)

This Spacelab 1 experiment was the first of a series of investigations designed by Nitsche and/or Schönholz et al. to study the low-gravity solution growth of a cadmium telluride crystal by the traveling heater method. The specific objective of the study was to produce a CdTe crystal free of gravity-induced defects and micro-inhomogeneities.

The Spacelab sample was composed of (1) a cylindrical seed crystal (18 mm length), (2) tellurium disk which acted as a solution zone (5 mm length), and (3) a CdTe feed rod (37mm length). The sample was configured within a quartz ampoule (110 mm length, 10 mm internal diameter). A quartz piston was also contained within the ampoule to insure volume minimization.

During the mission, the ampoule was inserted into the Spacelab 1 Mirror Heating Facility (MHF) and then evacuated. The zone was heated to 870 K and the ampoule adjusted by the Payload

Specialist. <Note: it was presumed that this adjustment was to place the zone within the optimum mirror focus.> The Te zone was then heated to 1070 K and held at that temperature for 1 hour. During this soak period the sample was rotated at 8 rpm. Translation of the furnace was then initiated (translation rate = 6 mm/day).

It was reported that, "Growth under low gravity lasted only 6 [hours] (instead of 17 [hours] as planned). In this time a layer of 1.5 mm (instead of 4.2 mm as planned) was deposited on the seed. Then the MHF was turned off by the controlling computer because of insufficient flow of coolant. The abrupt turn-off of the MHF also upset the cooling program. Instead of controlled temperature reduction from 1070 to 870 K within 32 min, the sample was practically quenched. The resulting thermal stress caused a close-meshed net of (110) cleavage cracks in the grown CdTe layer." (1, p. 164) <Note: Additional details of the MHF performance can be found in Reference (5).>

Reportedly, because of the unplanned furnace shutdown, the low-gravity processed material "...could not be analyzed satisfactorily." (1, p. 163) (See Reference (1) for brief comments concerning the sample appearance.)

<Note: Although it appeared that this experiment could be included in the "Crystal Growth From the Melt" chapter, responses from other investigators involved in travelling heater crystal growth experimentation indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Melt and Solidification, Metallic Solutions, Solution Zone, Binary Systems, Seed Crystals, Feed Material, Single Crystals, Sample Rotation, Rotating Fluids, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Thermosolutal Convection, Thermal Soak, Quench Process, Solid/Liquid Interface, Translation Rate, Crystal Homogeneity, Crystal Morphology, Crystalline Defects, Defect Density, Surface Morphology, Cracks, Piston System, Volume Compensation, Halogen Lamps, Vacuum, Hardware Malfunction, Processing Time Not As Long As Planned, Electronic Materials

Number of Samples: one
Sample Materials: cadmium telluride
(Cd*Te*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
CdTe is a material used in nuclear detection devices. When produced on Earth, the crystals usually contain a large number of defects which are detrimental to the detector performance. These defects are partially attributed to gravity-driven convective flows (buoyant or thermosolutal). It was expected that under low-gravity conditions, crystals containing fewer defects could be produced.

References/Applicable Publications:
(1) Schönholz, R., Dian, R., and Nitsche, R.: Solution Growth Of Cadmium Telluride Experiment ES 322. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 163-167. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48, Teledyne Brown Engineering publication. (acceleration measurements on Spacelab 1)

(3) Siffert, P., Biglari, B., Samimi, M., Hage-Ali, M., Koebel, J. M., Nitsche, R., Bruder, M., Dian, R., and Schönholz, R.: Characterization of CdTe Crystals Grown Under Microgravity Conditions. In Nuclear Instruments and Methods in Physics Research, A283, 1989, pp. 363-369. (post-flight; Spacelab D1 mission results)

(4) Eyer, A., Nitsche, R., and Zimmerman, H.: A Double Ellipsoid Mirror Furnace for Zone Crystallization Experiments in Spacelab. Journal of Crystal Growth, 47(1979), pp. 219-229. (furnace setup)

(5) Nitsche, R.: The Mirror Heating Facility: Performance and Experiences. Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 155-156.

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Co-Investigator(s): The Alabama Space and Rocket Center (2) <Note: The Alabama Space and Rocket Center is now called The U.S. Space and Rocket Center>, Buckbee, E. O. (Customer) (3)
Affiliation(s): (1) Prior to STS Launch: University of Alabama, Huntsville, Alabama, Currently: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (2) Huntsville, Alabama; (3) The Alabama Space and Rocket Center, Huntsville, Alabama

Experiment Origin: USA
Mission: STS Launch #13, STS-017 (STS 41-G Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: When originally proposed: College Student Experiment; NASA Get Away Special (GAS) Canister G-007
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-007: The Alabama Space and Rocket Center, Huntsville, Alabama
Processing Facility: Plexiglas™ cell fitted with two platinum electrodes
Builder of Processing Facility: University of Alabama, Huntsville, Alabama

Experiment:
Growth of Potassium-Tetracyanoplatinate Hydrate Crystals in an Aqueous Solution

This experiment was one of four investigations housed within the G-007 Getaway Special Canister during STS-017. (One other experiment (of the four) is applicable to this data base (see Henderson, STS-017 (Chapter 14)).) The experiment was the first in a series of investigations designed by Lee et al. to study the low-gravity, electrochemical growth of a single, complex inorganic crystal. The specific objective of the experiment was to grow single crystals of potassium tetracyanoplatinate hydrate (KCN) in an aqueous solution.

The crystal growth was to take place within a (6-ml volume), Plexiglas™ electrolysis cell configured with two small platinum electrodes. The experiment was to be initiated "...At the first available low-g period lasting 4 hours or more... [when a microprocessor powered] the electrolysis cell by a 1.3 V DC power supply...." (1, p. 112) This d.c. power supply furnished "...the potential across the platinum electrodes for nucleation of crystal growth" (1, p. 114) and crystals were expected to form on the anode. "A 35 mm camera and its electronic flash... [was to] have been activated at the same time, and... [was] to take a picture

every 40 minutes...." (1, p. 112) Miniature heaters were added to maintain the experiment at a constant temperature. The experiment required 24 hours of run time.

It was reported "...that because of an operational error... [G-007] was not activated as scheduled. Consequently, no experimental data were acquired. A careful postflight analysis revealed no evidence of anomalies within the cannister [sic] and plans were developed for a reflight on a subsequent shuttle mission." (5, p. 225) (See Lee, STS-032 for details concerning the subsequent shuttle experiment.)

Key Words: Crystal Growth From Solution, Inorganic Crystallization, Aqueous Solutions, Single Crystals, Electrolysis, Electrochemical Growth, Electric Field, Anode, Electrodes, Solid/Liquid Interface, Nucleation, Contained Fluids, Superconductivity, Linear Conductors, Sample Not Processed as Planned

Number of Samples: one
Sample Materials: potassium-tetracyanoplatinate hydrate (K*Pt*C*N*H*O*)
Container Materials: Plexiglas^{TM} cell

Experiment/Material Applications:
See Lee, STS-032.

References/Applicable Publications:
(1) Henderson, A. J., Jr.: Project Explorer: Getaway Special #007. In Get Away Special Experimenter Symposium, NASA Goddard Space Flight Center, August 1-2, 1984, pp. 111-117. (preflight)

(2) Henderson, A. J., Jr.: Project Explorer's Unique Experiments: Get Away Special #007. In Get Away Special Experimenter Symposium, NASA Goddard Space Flight Center, October 8-9, 1985, pp. 125-131. (post-STS-017; pre-STS-032)

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(7) NASA STS 41-G Press Kit, October 1984. (preflight)

(8) Input received from K. K. Dannenberg (The U.S. Space and Rocket Center), July 1993.

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Co-Investigator(s): The Alabama Space and Rocket Center (2) <Note: The Alabama Space and Rocket Center is now called The U.S. Space and Rocket Center,> Buckbee, E. O. (Customer) (3)
Affiliation(s): (1) Prior to STS-032: University of Alabama, Huntsville, Alabama, Currently: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (2) Huntsville, Alabama; (3) The Alabama Space and Rocket Center, Huntsville, Alabama

Experiment Origin: USA
Mission: STS Launch #24, STS-032 (STS 61-C, Columbia)
Launch Date/Expt. Date: January 1986
Launched From: NASA Kennedy Space Center, Florida
Payload Type: When originally proposed: College Student Experiment; NASA Get Away Special (GAS) Canister G-007R
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-007R: The Alabama Space and Rocket Center, Huntsville, Alabama
Processing Facility: Plexiglas™ cell fitted with two platinum electrodes
Builder of Processing Facility: University of Alabama, Huntsville, Alabama

Experiment:
Growth of Potassium-Tetracyanoplatinate Hydrate Crystals in an Aqueous Solution

This experiment was one of four investigations housed within the G-007 Get Away Special Canister during STS-032. (One other experiment (of the four) is applicable to this database (see Henderson, STS-032 (Chapter 14)).)

The experiment was the second in a series of investigations designed by Lee et al. to study the low-gravity electrochemical growth of a single, complex inorganic crystal (see Lee, STS-017). The specific objectives of the experiment included (1) the production of single crystals of potassium tetracyanoplatinate hydrate (KCN); (2) the investigation of "the governing reaction mechanism which... is thought to be one involving electron transfer; and [(3) the (hopeful) evaluation of] the electrical properties of the product as a 'linear chain conductor'." (5, p. 227)

A document released prior to the shuttle launch described the expected experimental setup. <Note: Details of the setup were not verified in post-flight documentation.> It appears that the crystal growth took place within a (6-ml volume) Plexiglas™

electrolysis cell configured with two small platinum electrodes. The cell was to be filled with 0.3 M concentration solution. A thermistor attached to the electrolytic cell was to monitor temperature fluctuations.

During the experiment, the "...electrochemical growth of single crystals of... (KCN) in an aqueous solution under the action of a small (1.3-volt dc) potential" was investigated (5, p. 224). The potential was placed across the electrodes, initiating crystal growth on the anode. Reportedly, the growth chamber temperature was to be regulated at 20 ^{o}C (+/- 10 ^{o}C). (A document published prior to the flight indicated that several small heaters were available to maintain the required temperature should the temperature fall below 10 ^{o}C.) The growth process was documented by intermittent flash photographs.

<Note: Although prior to the shuttle flight it was reported that "...crystal growth... [was] to begin when the best micro-gravity conditions exist[ed and] At the first available [low-gravity] period lasting 4 hours or more...," (2, p. 127) post-flight documentation did not indicate if this timing was achieved.>

Reportedly, "Several primary payloads on this Shuttle mission failed [not including this one], so the mission was terminated early to avoid a potential weather problem at landing. This GAS can was commanded off early after 3 days into the mission during deorbit preparations, and thus some experiment objectives were impacted. The nominal plan was 5 days of operation. As it turns out, the orbiter remained in orbit another 2 days because of the weather anyway." (7, p. 36)

Post-flight analysis of the payload indicated that the "...process performed quite successfully: four fine crystals, averaging about 2 millimeters in length, were formed as a result of the electrochemical action. These products actually represent two periods of growth, the first corresponding to the planned period of 24 hours, and a second 'bonus' period of 10 hours which arose after the first attempt at [shuttle] landing was waved off. Although the crystals are... much smaller than what would be expected for Earth-like-conditions, the growth process was demonstrated. This position is confirmed with the high quality photographs taken during the mission. The orbital environment was distinctly colder than that anticipated in advance, and this factor is most likely the major cause of the smaller size of the product. The on-off thermal cycling may also have influenced the results." (5, p. 228) Reportedly, the resultant crystals were too small to permit detailed examination.

Another report which described the post-flight analysis further detailed the cold temperatures experienced by the payload, yet seemed to contrast the information in Reference (5) (above). "At 10 hours and 10 minutes (10:10) the growth solution froze thus stopping all... [KCN] growth. The temperature had continually decreased from 5.6 ^{o}C at canister activation to -6.9 ^{o}C during this initial growth period.... The solution remained solid for the next 8 hours. A... photograph taken at 28:10 showed that the solution had liquefied. Finally at 43:10, the first crystals appear[ed] on the anode... after which the canister temperature once again dropped and the solution remained frozen for the rest of the mission.... The chamber temperature followed very closely to the canister temperature, indicating that the thermal control system was inadequate." (4, p. 49)

<Note: Reference (9), p. 92 indicated that (1) the heater batteries did not last and (2) the solution froze and thawed during the mission. It is not clear at what point in the growth of the crystals (the first growth period or the "bonus" growth period) that these events occurred.>

Key Words: Crystal Growth From Solution, Inorganic Crystallization, Aqueous Solutions, Single Crystals, Electrolysis, Electrochemical Growth, Electric Field, Anode, Electrodes, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Solid/Liquid Interface, Nucleation, Impurities, Crystalline Defects, Electrical Properties, Conductance, Contained Fluids, Battery Drain, Thermal Environment More Extreme Than Predicted, Freezing, Processing Time Not A Long As Planned, Superconductivity, Linear Conductors

Number of Samples: one cell
Sample Materials: potassium-tetracyanoplatinate hydrate (K*Pt*C*N*H*O*)
Container Materials: Plexiglas^{TM} cell

Experiment/Material Applications:
KCN was investigated for its applications as a "linear chain conductor." "These conductors have the interesting property of anisotrophy: their conductivity is different when measured along different directions in the solid." (4, p. 48)

"In recent years much attention has been given to the synthesis of linear conducting materials. These inorganic, organic, and polymetric materials have some very interesting electrical and optical properties, including low temperature superconductivity. The quest for methods to synthesize high quality single crystals of these highly conducting materials has been a very important part of previous research. Because of the anisotropic nature of these compounds, impurities and defects will strongly influence the unique physical properties of the most reproducible and purest crystals thus far. Space, specifically microgravity, eliminates phenomena such as buoyancy driven convection, and could permit formation of crystals many times purer than the ones grown to date." (4, p. 47)

References/Applicable Publications:

(1) Henderson, A. J., Jr.: Project Explorer: Getaway Special #007. In Get Away Special Experimenter Symposium, NASA Goddard Space Flight Center, August 1-2, 1984, pp. 111-117. (preflight)

(2) Henderson, A. J., Jr.: Project Explorer's Unique Experiments: Get Away Special #007. In Get Away Special Experimenter Symposium, NASA Goddard Space Flight Center, October 8-9, 1985, pp. 125-131. (post-STS-017; pre-STS-032)

(3) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, September 4, 1984. (short description; preflight)

(4) Cronise, R. J. IV: Electrochemical Growth of Linear Conducting Crystals in Microgravity. In NASA Goddard Space Flight Center's Get Away Special Experimenter's Symposium, October 27-28, 1987, NASA CP-2500, pp. 47-53. (post-flight)

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(8) NASA STS 61-C Press Kit, December 1985. (preflight)

(9) Stluka, E.: STS-61C Columbia Flight Final Report. In Goddard Space Flight Center's 1986 GAS Experimenter's Symposium, NASA CP-2438, pp. 87-93.

(10) Input received from K. K. Dannenberg (The U.S. Space and Rocket Center), July 1993.

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Co-Investigator(s): Unknown
Affiliation(s): (1) 3M Company, St. Paul, Minnesota

Experiment Origin: USA
Mission: STS Launch #14, STS-019 (STS 51-A, Discovery)
Launch Date/Expt. Date: November 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Diffusive Mixing of Organic Solutions (DMOS) cell (chemical reactors controlled by the Generic Electronics Module (GEM))
Builder of Processing Facility: 3M Company, St. Paul, Minnesota

Experiment:
Diffusive Mixing of Organic Solutions (DMOS-1)
Growth of Organic Crystals

To adequately investigate the nonlinear optical properties of materials, it is necessary to obtain single crystals of suitable size and quality. Various methods have been developed to produce such crystals on Earth including (1) gel diffusion and (2) frit-controlled diffusion. However, these techniques have been unable to produce the required high quality single crystals for non-linear optical property investigations. Gravity-induced effects such as sedimentation, convection, and turbulence are believed to be responsible for crystal defects (e.g., occlusions, dislocations) which reduce the quality of the material.

This STS-019 experiment was the first in a series of investigations sponsored by the 3M Company to study low-gravity, organic crystal growth. The specific objective of the experiment was to investigate the growth of urea and other organic crystals by chemical precipitation and Ostwald ripening.

The experiment was performed in six reactors. Reportedly, five of the chambers contained proprietary materials. Thus, only a single reactor designed to study the precipitation of urea crystals was discussed by the investigators.

The urea crystal growth experiment was performed in the Diffusive Mixing of Organic Solutions (DMOS) cell. The cell was contained in a NASA-supplied Experiment Apparatus Container (EAC). (See Reference (1) for a complete hardware description.) The urea reactor consisted of three chambers. Reportedly, "...an organic chemical solution [urea/methanol] is contained in one end chamber of the reactor. The solution is allowed to diffuse into the two remaining chambers [each containing toluene], which are filled with an incompatible solvent that causes the organic chemicals to

crystallize out of the solution." (5, p. 20)

Prior to opening the valves between the chambers, the cell and solutions were preheated for 5 hours. After preheating, the valves were slowly opened over another 5 hour period. (This valve-openning portion of the experiment took place during the crew sleep period such that the fluid system would experience a minimum disturbance with respect to g-level.) The valves remained open for the remainder of the flight (176 hours). During this period, the temperature of the cell was maintained at 30 ^{o}C.

Crystal collection by filtration occurred with 56 hours after the landing of the space shuttle. Reportedly, crystal growth caused by mixing occurred during and after re-entry to 1-g conditions.

Post-flight examination of the low-gravity processed urea materials revealed that the crystals had an average length of 8.16 mm and an average width of 0.48 mm. Terrestrial crystals had an average length of 6.08 mm and an average width of 1.10 mm. Average growth rates of the space-processed crystals were reported to be 0.0106 mm^3/hour, and average growth rates of the Earth-grown crystals were 0.0432 mm^3/hour. Generally, the Earth-grown crystals were thicker and contained more light-scattering centers than the low-gravity material. Seventy-eight percent of the crystals (1.64 gr) grown under low-gravity conditions were located in the central chamber, "...suggesting that crystal growth occurred when the urea solution diffused into toluene, but not vice versa. However, there was no information as to how much crystal growth occurred in the end chambers during and after [shuttle] re-entry.... In contrast, the ground-based results showed that virtually all of the crystal growth occurred in the end chamber containing the urea/methanol solution, suggesting that the initial diffusion of solutions and nucleation processes involved mechanisms which were different from the space-grown crystals." (1, p. 677)

The crystal yield for the low-gravity experiment was 30.29% versus 37.68% for the ground-based study. This difference may have been caused by the greater extent of mixing in the terrestrial solutions because of gravitational effects (e.g., convection caused by temperature and density gradients). Elemental analysis and x-ray powder diffraction experiments did not reveal significant differences (within experimental error) between the low-gravity and 1-g processed materials. However, shape-factor analysis (see Reference (1)) indicated that "...the shape of the space grown crystals is more needle-like, with a more narrow distribution." (1, p. 678)

Modeling of crystal growth rates and crystal morphology was performed using semi-empirical, quantum mechanical calculations (see Reference (1) for details). Comparison of these calculations to the experimental results led to the conclusion that "The preferred growth direction of urea is along the... [bar-4 axis]..., the direction along which hydrogen bonding is most significant, suggesting that urea crystal growth and crystal morphology is dominated by intermolecular interactions.... We conclude that crystal packing interactions must be considered in the future planning of crystal growth experiments in microgravity." (1, p. 679)

Key Words: Crystal Growth From Solution, Organic Crystals, Single Crystals, Model Materials, Solvent, Diffusion, Diffusive Mass Transfer, Double Diffusion, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Buoyancy-Driven Convection, Solutal Gradients, Density Difference, Sedimentation, Precipitation, Ostwald Ripening, Turbulent Flow, Growth Rate, Liquid/Liquid Interface, Solid/Liquid Interface, Crystal Morphology, Crystalline Defects, Dislocations, Needles, Occlusions, Three-Dimensional Crystalline Structure, Contained Fluids, Liquid Reservoir, Nonlinear Optical Materials, Optics Applications, Coated Surfaces, Acceleration Effects, Vehicle Re-entry Forces/Vibration

Number of Samples: Six reaction cells, although only the results from only one cell were reported.
Sample Materials: one cell: urea/methanol mixture with toluene solvent; other five cells: proprietary materials
Container Materials: Six stainless steel reactors, three of which were coated with Teflon™. <Note: The reactor used for the urea crystal growth experiment appears to have been coated with Teflon™.>

Experiment/Material Applications:
"Urea... is representative of one class of materials which are applicable to photonics, and served as a model compound and reference material in the DMOS experiment." (1, p. 673)

References/Applicable Publications:

(1) Gerbi, D. J., Egbert, W. C., Ender, D. A., Leung, P.C.W., Rochford, K. B., and Virden, J. W.: Growth of Organic Crystals in a Microgravity Environment. Journal of Crystal Growth, 76 (1986), pp. 673-680.

(2) 3M Experiment Diffusive Mixing of Organic Solids. 3M Fact Sheet, 3M Company, Public Relations Department, 225-5N (04) 3M Center, St. Paul, Minnesota 55144. (preflight)

(3) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 21-22. (post-flight)

(4) Gerbi, D. J., Ender, D. A., and Cook, E. L.: Nonlinear Optical Crystals Grown in Microgravity. Extended Abstracts, Nonlinear Optical Materials, Materials Research Society, edited by D.A.B. Miller, p. 129.

(5) NASA Press Kit, STS Mission 51-A, p. 20.

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Affiliation(s): (1) During STS-031: Space Research and Applications Laboratory, 3M Corporation, St. Paul, Minnesota, Currently: Corporate Research Laboratory, 3M, St. Paul, Minnesota; (2-8) 3M Corporation, St. Paul, Minnesota

Experiment Origin: USA
Mission: STS Launch #23, STS-031 (STS 61-B, Atlantis)
Launch Date/Expt Date: November 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
Processing Facility: Diffusive Mixing of Organic Solutions (DMOS-2) Cell
Builder of Processing Facility: 3M Corporation, St Paul, Minnesota

Experiment:
Diffusive Mixing of Organic Solutions (DMOS-2)

During the diffusive mixing of organic solutions in a quiet, low-gravity environment, (1) convective flows should be insignificant and (2) diffusive transport should prevail. Diffusive growth should produce crystals that are larger and of higher purity than crystals grown on Earth.

During a low-gravity space shuttle mission, crew and payload activities as well as spacecraft attitude maneuvers result in transient accelerations on the order of 10^{-3} g. Theoretical models have indicated that (1) these accelerations can result in convective flows and (2) these flows, in turn, may overwhelm the diffusive transport.

This STS-031 experiment was the second in a series of investigations sponsored by the 3M company to study low-gravity, organic crystal growth (see Egbert, STS-019 (this chapter)). The specific objectives of the experiment were to (1) characterize the dominant transport mechanisms active during the reduced gravity shuttle flight and (2) examine the effect of the shuttle gravity conditions on crystal growth.

During the STS-031 mission, the Diffusive Mixing of Organic Solutions (DMOS) apparatus was used to investigate (1) mixing of differing density solutions and (2) organic crystal growth. The apparatus, which was configured in a NASA-supplied Experiment Apparatus Container (EAC), contained six, three-chambered reactors. (The three-chamber design kept the reagents separate until the

experiment was activated.) A Generic Electronics Module (GEM) contained the data acquisition equipment as well as the controlling computer. (See Reference (2) for more details of the payload.) The chambers in each reactor were separated by motor-driven gate valves. These gate valves consisted of a slotted Teflon™ sliding plate sandwiched between two slotted stainless steel stationary plates. All reactor components which contacted the reagents were either made of Teflon™ or of Teflon™-coated stainless steel.

The DMOS-2 apparatus was similar to the DMOS-1 apparatus used during an earlier mission (see Egbert, STS-019) but had the following improvements: (1) a quick-change reactor installation capability, (2) larger chemical loading ports, (3) a valve-opening assembly that employed a lever instead of a roller and cam, and (4) electronics mounted on each cell (rather than a single control board for all six cells).

During the STS-031 experiment, two of the six reactors were used for a mixing experiment. One of the reactors contained methanol solutions of basic yellow dye and methylene blue dye in the outside chambers with pure methanol in the middle chamber. This reactor was used to examine mixing of components with comparatively small density variations. The second mixing reactor contained red dye (disulfone magenta (DSM) dissolved in chloroform) in one end chamber with heptane in the other two chambers. This reactor was used to examine mixing of components with comparatively large density differences. (Some fluid properties of the solutions in the reactors can be found in the materials section below.) The two mixing-experiment reactors contained combinations which bracketed the crystal growth experiments in terms of concentration and density differences.

The remaining four reactors were dedicated to crystal growth experiments. Two reactors were designed to investigate the ordering of molecules under nearly ideal conditions. The two remaining reactors were used to investigate (1) the method by which crystals pack together and (2) how this packing affects their electro-optical properties.

Three hours after the launch of the STS-031, the experiment was activated by a crew member. The six reactors were slowly preheated for 4 hours to a temperature of 33 ^{o}C. It was reported that, subsequently, the temperature was maintained between 34 ^{o}C and 38 ^{o}C. Nine hours after experiment activation (and just before the first astronaut sleep period) the valve opening phase was initiated. As preprogrammed, the valves were not fully opened until several hours later to assure minimum crew activity during initial solution mixing.

For the next 130 hours, the solutions were allowed to mix during regular crew and orbital maneuver activities. At the end of this period, the valves of the two fluid dynamics (mixing) experiments were closed to isolate the contents of each chamber for post-flight analysis. Twelve hours later, the experiment was completely deactivated. It was noted that the valves for the four crystal growth experiments were left open to allow any unmixed solutions to completely mix during re-entry and landing.

Approximately 18 hours after landing, the experiment was removed and returned to a laboratory for analysis. At this time, the flight experiment was duplicated in the laboratory under similar thermal conditions.

The following paragraphs present the results and discussion from the two fluid dynamics (mixing) experiments and two of the crystal growth experiments. It appears that the results from the remaining two crystal growth experiments were not reported (in the available publications) because they employed proprietary materials.

Fluid Dynamics Experiments
Large Density Difference Mixing Reactor:

Concentration in this reactor of the chloroform was determined using gas chromatography and concentration of the DSM was determined by using UV-VIS spectroscopy. It was reported that extensive transfer of components occurred during the 130 hour experiment time. Calculation of the diffusion times (characteristic diffusion length of 6-12 cm) and estimated binary diffusivity of chloroform and heptane of 2 or 3 x 10^{-5} cm^2/s) indicated that for diffusive mixing, 400 to 1600 hours would be required to reach equilibrium. Further calculations indicated that more than 2000 hours would be required. The fact that the actual experiment time was considerably less than any of these calculated diffusive mixing times "...demonstrates unambiguously that convective transport far exceeded diffusive transport." (2, p. 584)

Small Density Difference Mixing Reactor:

UV-VIS spectroscopy analysis indicated that after 130 hours the basic yellow and methylene blue solutions remained largely unmixed. A partial mixing of the dyes in the center chamber was reported. Mole fraction versus distance data demonstrated a high degree of symmetry, illustrating the isotropic nature of a diffusion controlled process. Ground-based experiments revealed com-

plete mixing at the end of 130 hours. One-dimensional diffusion flux calculations (diffusivity D = 0.87 x 10^{-5} cm^2/s for the yellow dye and D = 0.79 x 10^{-5} cm^2/s for the blue dye) were in good agreement with low-gravity experimental results (see Reference (2) for details). "This confirms that the small density gradients arising from the dye addition led only to insignificant convective contributions to the transport." (2, p. 585)

First Crystal Growth Experiment
N, N'-dimethylbarbituric acid (DMBA) Crystal Growth:

This experiment was conducted using a countersolvent or dilution crystallization procedure: a chloroform solution of DMBA was exposed to a heptane countersolvent (see Reference (2) for details of procedures). The solutions had relatively large density differences and, therefore, mixing must have occurred because of orbiter accelerations. This result was confirmed in the large density difference, fluids dynamics experiment.

The total mass of crystals obtained from the space experiment was 89% of the total mass of crystals obtained during the ground-based investigation. The space crystals were narrower and had a higher aspect ratio than the 1-g crystals. This result was explained "...based on the anticipated differences between the space and Earth interfacial boundary layers or depletion zones present in the nutrient solution around the growing crystals." (2, p. 587) (See Reference (2) for details.) The reduced convection during the low-g experiment did not produce crystals of superior size, purity, or optical uniformity when compared to the Earth crystals. Both 1-g and low-g crystals contained solvent inclusions and the melting points of both materials had similar sharpness. Proton nuclear magnetic resonance and powder x-ray studies revealed no differences.

Second Crystal Growth Experiment
TetraThiaFulvalene-TetraCyaNoQuinodimethane (TTF-TCNQ) Crystals:

The two end chambers of this growth reactor designated as chambers (A) and (C) contained acetonitrile solutions of TCNQ (0.0147 molar, chamber (A)) and TTF (0.0132 molar, chamber (C)). The center chamber (B) contained acetonitrile. The density differences between the solutions and the solvent were about 0.02 g/ml, and, therefore, the convective contributions should have been approximately the same as that observed in the yellow and blue dye mixing experiment.

Two different types of crystals were recovered from the low-g experiment: (1) planar blades or platelets (20 mm x 3 mm, 24.4 mg) and (2) microcrystalline needles (293 mg). The 1-g experiment produced only needles which indicated that the needles from the flight experiment were probably produced during the landing of the shuttle. The few large crystals obtained during space processing were much larger than those obtained on Earth. This result was attributed to the dominance of diffusive transport under low-gravity conditions. However, the low-g crystals did not possess a high degree of perfection: they were not of uniform shape nor did they have sharp, well-defined edges. The normalized conductivity measurements indicated that the Earth-grown crystals were of higher quality. It was also reported that x-ray diffraction studies indicated that the 1-g and low-g materials were crystallographically equivalent. Elemental analysis also detected no differences.

It was concluded that convective mixing occurred in those experiments involving fluids with large density differences. However, for those slight density difference, diffusive mixing was dominant despite the milligravity to microgravity accelerations present during the mission. Regardless of the active transport mechanism, the space crystals were not superior to those produced on Earth.

Key Words: Crystal Growth From Solution, Organic Crystals, Single Crystals, Solvent, Mass Transfer, Diffusion, Diffusive Mass Transfer, Double Diffusion, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Liquid Mixing, Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Solutal Gradients, Density Difference, Sedimentation, Precipitation, Growth Rate, Liquid/Liquid Interface, Solid/Liquid Interface, Ostwald Ripening, Crystal Morphology, Crystalline Defects, Sample Purity, Inclusions, Needles, Platelet Habit, Three-Dimensional Crystalline Structure, Contained Fluids, Liquid Reservoir, Nonlinear Optical Materials, Coated Surfaces, Acceleration Effects, Vehicle Re-Entry Forces/Vibration

Number of Samples: six, three-chambered cells
Sample Materials: First mixing reactor (small density variation): methanol solutions of basic yellow dye (3.1×10^{-3} molar, density at 20 ^{o}C of 0.79133 g/ml) and methylene blue dye (8.9×10^{-4} molar, density of 0.79087 g/ml) in the outside chambers with pure methanol (density of 0.79090 g/ml) in the middle chamber. Second

mixing chamber (large density variation): red dye (disulfone magenta (DSM) dissolved in chloroform, 6.2×10^{-4} molar) in one end chamber with heptane in the other two chambers. See experiment summary for the contents of the other reactors; proprietary materials.
Container Materials: All reactor components which contacted the reagents were either made of Teflon™ or of Teflon™-coated stainless steel. A Teflon™ sliding plate was also employed.

Experiment/Material Applications:
Materials processing in a reduced gravity environment may require strict diffusion control, or may take advantage of mass and thermal transport afforded by small accelerations present in low-Earth orbit. It is necessary to consider acceleration sources, mass and heat transport characteristics, and acceleration sensitivity of the experimental system in order to achieve the desired diffusion-dominated research goals.

The specific reasons why the sample materials were used were not presented in the available publications.

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(2) Radcliffe, M. D., Steffen, J. E., Cook, E. L., Cutting, J. F., Miller, L. R., Drake, M. C., Schroder, F. S., and Stevens, J.: Organic Crystal Growth in Low Earth Orbit. Journal of Crystal Growth, 1988, 92, pp. 581-590.

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Experiment Origin: USA
Mission: STS Launch #17, STS-024 (STS 51-B, Spacelab 3: Challenger)
Launch Date/Expt. Date: April 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Double Rack
Processing Facility: Fluid Experiment System (FES)
Builder of Processing Facility: TRW, Redondo Beach, California

Experiment:
Fluid Experiment System (FES) Growth of Crystals in Solution (MPS770058)

When crystals are grown from solution in a terrestrial laboratory, small density differences in the fluid surrounding the growing crystal (due to either localized temperature or concentration variations) give rise to steady, laminar buoyancy-driven convection in the fluid.

"...vigorous stirring is used in order to increase the rate at which crystals can be grown without the formation of solvent inclusions. Inclusions are the most significant defects in crystals grown from solution, especially for optical applications. While it is known that buoyancy-driven convection is inferior to forced convection, it is not known whether the elimination of convection is desirable or not....

"In a low-g environment, convection is greatly suppressed and diffusion becomes the predominant mechanism for thermal and mass transport. In the absence of convection, crystal growth from solution will rapidly become slower as the adjacent solution is depleted in solute, unless the growth temperature is lowered to compensate. However, if the temperature is lowered too fast the growth rate will reach the level at which solvent inclusions are formed." (9, p. 1)

This Spacelab 3 experiment was designed to study the growth of single triglycine sulfate crystals from solution in the low-gravity environment. The specific objectives of the research were "...(1) to develop a technique for solution crystal growth in a low gravity environment, (2) to characterize the growth environment provided by an orbiting spacecraft and to determine the

influence of the environment on the growth behavior, and (3) to determine how gravity in a microgravity environment influences the properties of a resulting TGS crystal." (9, p. iii) The experiment employed a new crystal growth technique which allowed programmed heat extraction from the seed crystal.

The experiment was performed in the Spacelab 3 Fluids Experiment System (FES). The FES was specifically designed to investigate fluid phenomena using optical observation techniques. The system consisted of (1) an interchangeable crystal growth experimental cell and (2) a support module.

Three experiment cells were prepared for the mission. Each cell was filled with a solution of TGS above the saturation temperature. A sting assembly, affixed at one end of the cell, extended into the TGS solution. A TGS seed crystal, attached to the sting tip, was protected from the solution prior to the initiation of crystal growth by a cap assembly.

The support module included several components including (1) an optical bench with holographic facility, (2) a schlieren optical setup, (3) control electronics, and (4) three-axis accelerometers.

Three experimental runs were planned for the Spacelab 3 mission. A run was initiated by placing the FES experiment cell into a preheat enclosure and raising the temperature of the solution to 70 ^{o}C. (At 50 ^{o}C a stirrer inside the cell was initiated to "dissolve the crystallites faster.") The FES remained at this temperature until the fluid loop of the optical bench was heated and and held at a temperature of 70 ^{o}C. The FES was then transferred to the optical bench, and the experiment temperature attained. <Note: This temperature was not specified.> The temperature was equilibrated first using a stirrer (later the stirrer was not used). After it was determined that (1) the solution was at the desired temperature above saturation and (2) there were no crystallites in the fluid, the cap covering the seed crystal was retracted, bringing the seed into contact with the TGS solution. Because the cell temperature was slightly higher than the saturation temperature, the outer surface of the crystal dissolved. The temperature was then slowly lowered to the desired growth temperature and stabilized. Crystal growth was initiated by extracting heat from the seed crystal through the thermoelectrically cooled sting thereby creating the desired undercooling near the growing crystal.

The cell and sting/seed temperatures were controlled by a microprocessor to an accuracy of +/- 0.1 ^{o}C and +/- 0.01 ^{o}C, respectively. The cell temperatures were monitored at three places: (1) on the sting base, (2) 1 cm away from the sting (in

the fluid), and (3) at the cell walls. Holograms of the experiment cell were obtained from two orthogonal directions using an He-Ne laser directed through the cell's primary and transverse optical windows. (The holograms provide a detailed picture of the fluid refractive index around the growing crystal, from which concentration data can be deduced.) The crystal growth was monitored onboard Spacelab and on Earth by the real-time video schlieron system.

At the end of the growth period the cap was placed over the crystal. A crew member then (1) removed the sting assembly from the cell, (2) allowed the crystal to cool, (3) dried the crystal, and (4) stowed the crystal for the mission back to Earth.

Reportedly, "Two crystal runs were successfully performed during the mission. In both [of these successful] experiment runs the starting seed... [cystals were] TGS crystal discs with (001) orientation, 3.42 mm thickness and 15.07 mm and 9.98 mm diameter, respectively. The first experiment run in which (010) oriented seed was planned to be used could not be completed due to some hardware problems. The second experiment run was performed for 58 hr where both sting/crystal and walls were cooled at a programmed rate, where T(substing) < T(subsol) < T(subsat). The third run was accomplished in 32.2 hr, where T(substing) = T(subsol) < T(subsat). The total average growth for both crystals... [was]... [approximately] 0.4 mm. The growth, however, was not quite uniform across the exposed face due to some apparent damage to seed prior to growth. There was less growth in the center of the seed...." (6, p. 19)

Post-flight studies included interferogram reconstruction as well as characterization of the space-processed crystals. The crystals were cleaved perpendicular to the (001) face to examine the quality of the (010) face for pyroelectric qualities. The following conclusions were reported:

(1) A diffusion-controlled growth was indicated by the lack of a visible interface between the seed and space grown crystal.

(2) Diffusion-controlled growth was also indicated by the reconstructed interferogram from the third experiment run showing an axially symmetric concentration field about the growing crystal. <Note: It was reported that the interferograms would be digitized to determine concentration and temperature fields around the crystal and that these data would be compared to a finite difference model.>

(3) "The normalized detectivity D^* (1000 K, 15 Hz, 1Hz) for space grown crystal No. 204 [run 3] for 3 mil thickness (1.0×10^8 W^{-1} cm $Hz^{1/2}$) is somewhat less than the best Earth grown crystal

detectors for sample thickness of 0.7 mil thickness (1.0×10^{9} W^{-1} cm $Hz^{1/2}$)." (6, p. 19)

(4) The good quality of the space-processed crystals was indicated by (a) the higher pyroelectric coefficient (about 43 nC/cm^2 ^{o}C) and (b) the hysteresis loop shape.

(5) The observed, low-gravity crystal growth rate agreed with the theoretically predicted growth rate with a diffusion coefficient of 2×10^{-5} cm^2/sec.

(6) Measurements from drifting crystallites indicated a quasi-steady-state acceleration level of 4×10^{-7}. Accelerometers attached to the FES also indicated vibrations of 10^{-3} g at frequencies of 10 Hz and above.

(7) Solution crystal growth by the cooled sting technique was successfully tested.

Reference (9) contains detailed discussions about many aspects of this experiment including (1) seed crystal characteristics at the initiation of crystal growth, (2) crystal characteristics after crystal growth (including electrical characterization, etc.), (3) post-flight observations of the experimental cell contents, (4) low-g accelerometer data analysis, (5) holographic analysis, and (6) computational modeling of the crystal growth.

<Note: Not all of the publications listed below were available to aid in the preparation of this summary.>

Key Words: Crystal Growth From Solution, Seed Crystals, Single Crystals, Transparent Liquids, Cooled Sting Technique, Density Difference, Thermal Gradient, Solutal Gradients, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Diffusion Coefficient, Heat and Mass Transfer, Thermosolutal Convection, Buoyancy-Driven Convection, Stirring of Components, Solvent, Inclusions, Saturation, Growth Rate, Undercooling, Thermal Equilibrium, Solid/Liquid Interface, Interface Phenomena, Interface Physics, Surface Morphology, Crystal Morphology, Crystalline Defects, Contained Fluids, Acceleration Measurements, Acceleration Effects, Liquid Vibration, Hardware Malfunction, Holography, Schlieren Viewing, Infrared Detector Applications, Optics Applications, Electronic Materials

Number of Samples: three
Sample Materials: triglycine sulfate (TGS), $(NH_2CH_2COOH)_3H_2SO_4$ (N*H*C*H*C*O*H*, H*S*O*)
Container Materials: Unknown. The cell wall material was not identified.

Experiment/Material Applications:
"TGS crystals were selected as a candidate growth material because they can be grown at comparatively low temperature (30 ^{o}C-45 ^{o}C); TGS solution is a transparent system so that holographic techniques could be employed to study fluid properties in order to characterize the growth environment; TGS has high technological importance for infrared detectors (8 to 14 μ) operating at room temperature; present devices have detectivities about an order of magnitude below the theoretically predicted values." (6, p. 18)

References/Applicable Publications:
(1) Lal, R. B., Kroes, R. L., and Wilcox, W. R.: Growth Of Triglycine Sulfate (TGS) Crystals by Solution Technique. In Materials Processing in the Reduced Gravity Environment of Space, Proceedings of the Annual Meeting, Boston, Massachusetts, November 16-18, 1981, pp. 399-408.

(2) Kroes, R. L. and Reiss, D.: Properties of TGS Aqueous Solution for Crystal Growth. Journal of Crystal Growth, 69, 1984, pp. 414-420. (ground-based research and properties of TGS)

(3) Spacelab 3 Early Results. Spaceflight, Vol. 27, 1985, p. 361. (post-flight)

(4) Naumann, R. J.: Microgravity Science and Applications Program in the United States. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), pp. 27-28. (post-flight)

(5) Owen, R. B. and Kroes, R. L.: Holography on the Spacelab 3 Mission. Optics News, Vol. 11, No. 7, pp. 12-16, 1985.

(6) Lal, R. B., Aggrawal, M. D., Batra, A. K., Kroes, R. L., Wilcox, W. Trolinger, J. R., and Cirino, P.: Growth of Triglycine Sulfate (TGS) Crystals Aboard Spacelab 3. Spacelab 3 Mission Science Review, Proceedings of a symposium held at NASA George C. Marshall Space Flight Center, Alabama, December 4, 1985, NASA CP-2429, pp. 18-26. (See also pp. 3-4 for short summary; post-flight)

(7) Morgan, S. H., Silberman, E., Kroes, R. L., and Reiss, D. A.: Raman Study of the Diffusion of Triglycine Sulfate in Aqueous Solutions. Appl. Spectro., Vol. 40, No. 1, pp. 35-38, 1986.

(8) Reiss, D., Kroes, R. L., and Anderson, E. E.: Growth Kinetics of the (001) Face of TGS below the Ferroelectric Transition Temperature. J. Crystal Growth, Vol. 84, pp. 7-10. 1987.

(9) Lal, R. B., Aggarwal, M. D., Batra, A. K., and Kroes, R. L.: Solution Growth of Crystals in Zero Gravity. Final Technical Report, Prepared by Alabama Agricultural and Mechanical University, Department of Physics, Huntsville, Alabama, NASA Contract No. NAS8-32945, July 1987, 218 pp. (post-flight; detailed description)

(10) Materials Processing in Spacelab 3. Application Payload Projects, Spacelab Payload Projects Office, Marshall Space Flight Center, Huntsville, Alabama. (preflight; FES description)

(11) Lal, R. B.: A Study of Crystal Growth by Solution Technique. NASA CR-3370, January 1981, 30 pp. (related research)

(12) Input received from Principal Investigator R. B. Lal, August 1988.

(13) Rogers, M.J.B. and Alexander, J.I.D.: A Strategy for Residual Acceleration Data Reduction and Dissemination. In Adv. Space Res., Vol. 11, No. 7, 1991, pp. 5-8. (post-flight, acceleration data)

(14) Rogers, M.J.B. and Alexander, J.I.D.: Residual Acceleration Data Analysis for Spacelab Missions. In Microgravity Science and Technology, 1992, pp. 43-49. (post-flight, acceleration data)

(15) Rogers, M.J.B. and Alexander, J.I.D.: Analysis of Spacelab 3 Residual Acceleration Data. In Journal of Spacecraft and Rockets, Vol. 28, No. 6, 1991, pp. 707-712. (post-flight, acceleration data)

<Note: See Reference (9) for an extensive listing of publications related to this experiment.>

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Co-Investigator(s): Izquierdo, M. (Project Engineer) (2)
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Experiment Origin: USA
Mission: STS Launch #18, STS-025 (STS 51-G, Discovery)
Launch Date/Expt. Date: June 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) Canister G-034
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-034: Texas High Schools (Ysleta and El Paso Districts)/Dickshire Coors, El Paso, Texas
Processing Facility: Chamber containing a pre-saturated solution of material which by a pressure releasing process would initiate crystallization
Builder of Processing Facility: Unknown

Experiment:
Crystallization of Potassium-Aluminum-Sulfate

This investigation was one of 13 experiments housed within the G-034 Get Away Special Canister. Three other of these 13 investigations are applicable to this data base (see Casarez, STS-025 (Chapter 18), Foster, STS-025 (Chapter 2), and Thurston, STS-025 (Chapter 18)). The overall objective of the research was to compare space-produced and terrestrial-produced potassium-aluminum-sulfate crystals.

Prior to the mission, a saturated solution of water and potassium-aluminum-sulfate was prepared and placed into a growth chamber. The expected inflight procedure was described as follows: "When the experiment is turned on, a linear actuator will push the plunger down a small shaft, thus relieving the negative pressure within the growth chamber. The crystals should then begin to grow." (1, p. 66) Reportedly, the temperature was to be maintained above 0 ^{o}C during the experiment.

During the mission, a Plexiglas case enclosing a seed germination experiment (another of the investigations configured within the can) broke "...spilling a water/formaldehyde mixture inside the GAS can. Several seconds later the batteries and/or controller shorted, ending all experiments except ...[a wicking of fluids experiment (See Foster, STS-025)] which had its own power supply and controller." (4, p. 34)

Further information describing the experimental setup and expected results could not be located at this time.

Key Words: Crystal Growth From Solution, Aqueous Solutions, Saturated Solution, Solid/Liquid Interface, Crystal Morphology, Liquid Leakage, Contamination Source, Piston System, Hardware Malfunction, Battery Short

Number of Samples: one
Sample Materials: potassium-aluminum-sulfate solution and water (K*Al*S*, H*O*)
Container Materials: unknown

Experiment/Material Applications:
"It is expected that better, or even close to perfect crystals will be grown <in the space environment>. They will probably be much clearer and may possibly have a different shape than those grown on earth." (1, p. 66)

References/Applicable Publications:
(1) El Paso & Ysleta Schools Get Away Special Payload #34. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, August 1-2, 1984, NASA CP-2324, pp. 59-68. (preflight)

(2) Cargo Systems Manual: GAS Annex for STS 51-G, JSC 17645 51-G, Rev. A, March 20, 1985. (very short description; preflight)

(3) NASA Space Shuttle Mission 51-G Press Kit, June 1985, p. 20. (very short description; preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special Canister mission history)

(5) G-034 Payload Accommodations Requirements, NASA Goddard Space Flight Center, 1985.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, MEDEA Double Rack
Processing Facility: Monoellipsoidal Mirror Furnace (ELLI)
Builder of Processing Facility: Dornier System GmbH, Germany

Experiment:
Growth of PbSnTe by Travelling Heater Method (MD-ELI-02)

This Spacelab D1 experiment was designed to investigate the processing of a PbSnTe alloy by the Traveling Heater Method (THM). <Note: A discussion of the THM method can be found under Nitsche, Spacelab 1 (this chapter).> The specific objectives of the study were to investigate how the crystalline properties were influenced by (1) low-gravity THM processing and (2) gravity-driven convection in the THM solution zone.

The cylindrical 15-mm-diameter Spacelab sample consisted of three parts: (1) an 8-mm long, Bridgman-grown $Pb_{0.8}Sn_{0.2}Te$ seed crystal, (2) an 8-mm long $Pb_{0.62}Sn_{0.27}Te_{0.11}$ solution zone, and (3) a 13-mm long $Pb_{0.8}Sn_{0.2}Te$ feed crystal. These three sections were sandwiched between two graphite blocks. The lower graphite block (18 mm long) contained two thermocouples. The assembly was configured within a quartz ampoule such that a small slit existed between the ampoule wall and sample material. A volume compensation mechanism was included in the upper graphite block.

During the mission, the sample was processed in the ELLI Monoellipsoid Mirror Furnace. The sample was placed in the furnace and power supplied to the lamps until the desired temperature was reached. This temperature was maintained for a few hours to allow homogenization of the solution zone. The sample was then rotated (10 rpm) while being pulled at a rate of 3.7 mm/day. A total growth length of 2.8 mm was achieved. (The complete time/temperature profile is included in Reference (2)). For comparison, a ground-based reference experiment was performed using the same sample setup and time-temperature profile.

Post-flight examination of the sample included (1) visual and metallographic inspection, (2) Energy Dispersive X-ray Analysis (EDAX), (3) x-ray diffraction, (4) infrared reflection measurements, (5) Hall effect measurements (Van-der-Pauw Method), (6)

electrochemical etching, and (7) element selective etching.

Visual inspection of the low-gravity sample revealed growth facets and etch pits (before application of etchant) on the surface. The etch pits were attributed to selective evaporation from the surface into the slit area between sample and ampoule wall, resulting in gas phase etching. The metallographic examination (sample cut axially) revealed two spherical shrink holes, one touching the growth boundary of the single crystal. A 1-g processed sample contained a ring-shaped shrink hole around the surface of the solution zone. The differences between the shrink holes of these two samples was attributed to the lack of convection in the low-gravity sample.

EDAX examinations revealed three distinct types of inhomogeneities (axial tin profiles):

(1) Long range inhomogeneity: A systematic shift in tin concentration was found in the 1-g sample but not the low-gravity sample.

(2) Medium range inhomogeneity: An oscillation of tin content (period = about 1 mm) in (a) the Bridgman-grown seed material of both 1-g and low-gravity processed samples, (b) the THM grown portion of the 1-g sample, and (c) the first 1 mm of the THM portion of the low-gravity processed sample (after which the oscillation disappeared).

(3) Short-range variations: Noise-like variation of tin content in both the low-gravity and 1-g samples.

Hall effect measurements indicated a significant increase in charge-carrier density for the low-gravity sample (THM grown material). In all Earth-processed samples, the maximum charge-carrier density was 1900 cm^2/Vs. The sample processed during the Spacelab D1 mission had a charge-carrier density of 5500 cm^2/Vs. "A possible explanation of this difference might be the absence of stresses (on ground induced by contact with the ampoule wall) has reduced the number of scattering centers for the charge carriers in the... [low-gravity]... sample." (2, p. 286) (Infrared reflectivity measurements allowed carrier density determinations over the axial cut. These results may be located in Reference (2).)

<Note: Although it appeared that this experiment could be included in the "Crystal Growth From the Melt" chapter, other investigators involved in travelling heater method experimentation indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Metallic Solutions, Solution Zone, Ternary Systems, Seed Crystals, Feed Material, Single Crystals, Sample Homogeneity, Sample Rotation, Pulling Rate, Mass Transfer, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Solid/Liquid Interface, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Etch Pits, Facets, Volume Compensation, Volume Change, Stress, Crucible Effects, Evaporation, Gas Leakage, Lamps, Semiconductors, Semiconductor Applications, Infrared Detector Applications, Electronic Materials

Number of Samples: one
Sample Materials: Composition of material to be grown: $Pb_{0.8}Sn_{0.2}Te$; solution zone composition: $Pb_{0.7}Sn_{0.3}Te_{0.11}$ (Pb*Sn*Te*)
Container Materials: quartz ampoule
(Si*O*)

Experiment/Material Applications:
Lead-tin-telluride (Pb_{1-x}-Sn_x-Te) is a semiconductor material used for infrared detectors and infrared lasers. Its optical and electronic properties depend on the ratio of Pb to Sn.

References/Applicable Publications:
(1) Harr, M. and Brunsmann, U.: Growth of PbSnTe by the Travelling Heater Method. In Scientific Goals of the German Spacelab Mission D1, German publication, WPF, 1985, pp. 119-120. (preflight)

(2) Haar, R., Dornhaus, R., and Brotz, G.: Growth of PbSnTe by the Travelling Heater Method. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 283-288. (postflight)

(3) Schönholz, R., Dian, R., and Nitsche, R.: Growth of Cadmium Telluride by an Improved Travelling Heater Method. In Journal of Crystal Growth, Vol. 72, 1985, pp. 72-79. (discussion of THM method)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: Radiation from halogen lamps is focused on sample by ellipsoidal mirrors
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Federal Republic of Germany

Experiment:
Growth of Cadmium Telluride from a Liquid Phase (Te Solution) by the Travelling Heater Method (WL-MHF 03)

This Spacelab D1 experiment was the second of a series of investigations designed by Schönholz and/or Nitsche et al. to study the low-gravity solution growth of cadmium telluride by the travelling heater method (see Nitsche, Spacelab 1). The specific objective of this investigation was to produce a CdTe crystal free of gravity-induced defects and micro-inhomogeneities.

An internally graphitized ampoule contained (1) a cylindrical CdTe seed crystal (9.75 mm diameter, 9.9 mm long), (2) a Te solvent zone (9.70 mm diameter, 8.1 mm long), and (3) a CdTe feed rod (9.7 mm diameter, 34.2 mm long). "The material was chlorine doped to about $10^{17}/cm^3$ by addition of $CdCl_2$." (3, p. 281)

During the mission, the ampoule was inserted in the Spacelab Mirror Heating Facility (MHF). The facility "...was brought to 'observation temperature' (lamp power P=44 W) to enable fine adjustment by the payload specialist." (3, p. 281) <Note: it was presumed that this adjustment was to place the zone within the optimum mirror focus.> Heater power was then increased to 81 W within 6 minutes and the solvent zone was equilibrated for 125 minutes. Furnace translation was initiated (5 mm/day) and crystal growth proceeded for 21 hours. Finally, the sample was annealed by (1) reducing lamp power to 46 W and (2) holding it for 25 minutes at this level. The furnace was then switched off.

It was reported that problems arose during the experimental procedure. "Although the experiment was nominally carried out according to schedule, its outcome was considerably hampered by serious deviations from the planned conditions:

"1. The lamp power of the MHF, monitored by a photo cell, was too low: 81 W instead of 115 W in the calibration experiments. (Possible reasons: use of different lamp model or defective photocell). <Note: Presumably the calibration experiments refer to 1-g reference tests performed prior to the mission.>

"2. Photographs taken during the mission showed that the foci of the lamps were asymmetric in respect to the ampoule. (Possible reasons: Maladjustments of lamps and/or sample).

"3. The mechanism for rotating the ampoule was not working. Thus the solvent zone and the growing interface were exposed to the asymmetric temperature field of the MHF." (3, p. 281)

The failures described above resulted in (1) an asymmetric solution zone (warm and cold sides) resulting in unexpected growth conditions and (2) a reduced zone temperature which caused (a) a reduced solute concentration in the Te solvent, (b) a smaller zone length, and (c) a reduced solute transport rate.

Reference (3) stated "Because of too low a temperature setting and failure of the rotation mechanism, unfavourable growth conditions prevailed during the experiment. Thus no definite conclusions can be drawn on a beneficial action of a microgravity environment in this system." (3, p. 283) However, a later publication (Reference (5)) included analysis and conclusions from this experiment. A discussion of these results is presented below.

Comparison of the space-grown sample with a similarly processed Earth-grown sample (heater power = 116 W, no rotation) demonstrated better performance of the low-gravity processed material. The concentration of electrically active defects was less for the space-processed sample. The etchpit density of the space sample ($1 \times 10^2/cm^2$) was lower than the Earth-processed sample ($5 \times 10^2/cm^2$). The photoconductivity and resistivity studies (which characterize carrier transport properties) of the low-gravity processed specimen indicated an improvement over the Earth-processed sample. These results were attributed to a smaller defect concentration in the space sample (see Reference (5) for details).

<Note: Although it appeared that this experiment could be included in the "Crystal Growth From the Melt" chapter, other investigators involved in travelling heater method experimentation indicated that the appropriate chapter for the experiment was

"Crystal Growth From Solution".>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Metallic Solutions, Solution Zone, Solvent, Binary Systems, Ternary Systems, Dopant, Seed Crystals, Feed Material, Single Crystals, Sample Rotation, Translation Rate, Mass Transfer, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Thermosolutal Convection, Asymmetric Temperature Field, Annealing, Thermal Equilibrium, Concentration Distribution, Solid/Liquid Interface, Crystal Homogeneity, Crystal Morphology, Crystalline Defects, Defect Density, Surface Morphology, Coated Surfaces, Halogen Lamps, Processing Difficulties, Furnace Malfunction, Electronic Materials

Number of Samples: one
Sample Materials: cadmium telluride doped with chlorine (Cd*Te*Cl*)
Container Materials: internally graphitized quartz (Si*O*)

Experiment/Material Applications:
See Nitsche, Spacelab 1.

References/Applicable Publications:
(1) Schönholz, R., Dian, R., and Nitsche, R.: Growth of Cadmium Telluride by the Travelling Heater Method (THM) from Tellurium Solution (Expt: D1-WL-MHF 03). In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 61. (abstract only; post-flight)

(2) Schönholz, R. and Nitsche, R.: Growth of Cadmium Telluride by the Travelling Heater Method form Tellurium Solution. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 117-118. (preflight)

(3) Dian, R., Schönholz, R., and Nitsche, R.: Growth of Cadmium Telluride by the Travelling Heater Method From Tellurium Solution. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 280-283. (post-flight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements on D1)

(5) Siffert, P., Biglari, B., Samimi, M., Hage-Ali, M., Koebel, J. M., Nitsche, R., Bruder, M., Dian, R., and Schönholz, R.: Characterization of CdTe Crystals Grown Under Microgravity Conditions. In Nuclear Instruments and Methods in Physics Research, A283, 1989, pp. 363-369. (post-flight; also discusses results from Bruder, Spacelab D1 (Chapter 10))

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Experiment Origin: Belgium
Mission: TEXUS 13
Launch Date/Expt Date: April 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-8: furnace with eight sample chambers
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Crystal Growth from Solution

This TEXUS 13 experiment was designed to study low-gravity solution crystal growth. Reportedly, the specific objective of the study was to qualitatively investigate variations in crystal parameters before and after processing in low gravity. <Note: The specific parameters of interest where not detailed in the available publications.>

An eight-chambered "kiln", housed within TEXUS Experiment Module TEM 06-8, was employed for the experiments. Prior to the rocket launch, eight samples were configured in the kiln. (See the **Sample Materials** section (below) for specific compositions of each of the samples.)

Just prior to rocket lift-off, the samples were heated to 270 ^{o}C. During the first 75 seconds of the rocket's low-gravity phase, the temperature was held at 270 ^{o}C. During the subsequent 400 seconds of the low-gravity phase, the samples were cooled to 40 ^{o}C by a freon cooling system.

Post-flight, the low-gravity samples were compared to similarly processed terrestrial samples. Analysis indicated that the low-gravity and 1-g sample x-ray diffraction patterns were identical for the specimens $AgNO_3$, $LiNO_3$, HBO_2, and $HgCl_2$. However, "Well pronounced differences were observed for HgI_2, $HgBr_2$, and the two eutectic mixtures:

"It is characteristic that there are less X-ray reflections for the samples crystallized under microgravity condition.

"It is not excluded that the possible decompositions occur at 270 ^{o}C. In order to ascertain the value of the obtained results and to calculate the lattice parameters of the single components, more experiments with the same rate of freon cooling under gravity condition at different temperatures are required. If decomposition does not occur it is clear that another crystallographic phase will be formed under microgravity conditions." (1, p. 44)

No further discussion of this experiment could be located.

<Note: Although it appeared that this experiment could be included in the "Crystal Growth From the Melt" chapter, Vochten indicated that the appropriate chapter for the experiment was "Crystal Growth From Solution." It is suspected, but not verified, that the experiment may have employed the travelling heater method.>

Key Words: Crystal Growth From Solution, Travelling Heater Method, Melt and Solidification, Solution Zone, Eutectics, Ternary Systems, Binary Systems, Diffusion, Solid/Liquid Interface, Crystal Homogeneity, Crystal Morphology, Surface Morphology

Number of Samples: eight
Sample Materials: (1) $AgNO_3$, (2) HgI_2, (3) $HgBr_2$, (4) $HgCl_2$, (5) HBO_2, (6) $LiNO_3$; eutectic mixtures: (7) 0.52 mol% $HgBr_2$ and 0.48 mol% $HgCl_2$, (8) 0.41 mol% HgI_2 and 0.59 mol% $HgBr_2$
(Ag*N*O*, Hg*I* Hg*Br*, Hg*Cl*, H*B*O*, Li*N*O*)
Container Materials: unknown <Note: Although the Principal Investigator indicated what this material was, his hand-written entry was illegible.>

Experiment/Material Applications:
The Principal Investigator reported that the sample materials were selected because of their melting points.

The specific research application of this experiment was not detailed in the available publications.

References/Applicable Publications:

(1) Vochten, R.: Crystal Growth from Solution. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 44. (post-flight)

(2) Crystallisation[sic] Experiments with Salts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 190. (post-flight)

(3) Input received from Principal Investigator R. F. Vochten, September 1993.

(4) Experimentmodul TEM 06-8. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 41. (post-flight; module description)

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Co-Investigator(s): Saturnelli, A. M. (Teacher/Advisor) (2), Union College (Sponsor) (3), Scaife, C. W. (Experiment Advisor) (4)
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Experiment Origin: USA
Mission: STS Launch #26, STS-26 (Discovery)
Launch Date/Expt. Date: September 1988
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment, Shuttle Student Involvement Program (SSIP), Middeck Experiment
Processing Facility: Three chemical reactors.
In each of the chemical reactors, a solution of lead acetate was separated from a solution of potassium iodide by a chamber containing (1) water and (2) a semi-permeable membrane.
Builder of Processing Facility: Pierson, R., and O'Rourke, J., Engineering Machine Laboratory, Union College, Schenectady, New York

Experiment:
Utilizing a Semi-Permeable Membrane to Direct Crystal Growth (SE82-5)

This STS-26 student experiment was designed to investigate the "double-displacement-reaction" solution crystal growth of lead iodide. The major objectives of the research were to (a) control the crystal growth using a semi-permeable membrane and (b) produce large, pure, single crystals of lead iodide. It was speculated that the rate of diffusion through the membrane and the initial concentrations of the lead acetate and potassium iodide solutions could dictate which side of the membrane crystals grew, the growth rate, the size, and the shape of the final crystals.

Prior to the shuttle launch, three experiment reactors were prepared. In each reactor, a solution of lead acetate was separated from a solution of potassium iodide by a chamber containing water. This inner water chamber was divided down the middle by a semi-permeable, cellulose membrane (template).

During the STS-26 mission, valves isolating the inner chambers were opened, permitting the diffusion of the solutions toward the membrane. As the solutions came into contact, a double replacement reaction took place: "[The] Lead acetate and potassium iodide... [reacted] to form insoluble lead iodide crystals, potassium ions, and acetate ions. As the ions... [traveled] across [the] semi-permeable membrane, the lead and iodide ions... [collided and formed] the lead iodide crystals." (4, p. 36) "Video recordings of the 40-hour experiments show[ed] crystals beginning to form on the membrane 30 to 120 seconds later." (3, p. 206)

The space crystals were compared to terrestrial-grown crystals. Video tape images of the growth process indicated that while on Earth, crystals form only on the lower half of the membrane; the space grown crystals form (1) over the entire membrane and (2) in the solution away from the membrane. Reportedly, crystals in both the Earth and space experiments grew "...on the side of the membrane initially containing the lead ions, therefore, iodide ions are migrating through the cellulose membrane." (7, p. 11)

Purity, size, crystal color, density, hardness, morphology, refractive index, and electrical and thermal characteristics were examined. Reportedly, the space-grown crystals were better insulators and exhibited higher analytical purity in terms of foreign elements. (9) "For unknown reasons, significantly higher levels of carbon appear to have migrated out of the growth reactors on Earth... Shuttle-grown crystals contained only about one-fifth the carbon contamination of those produced on Earth." (3, p. 206) "In addition, Earth-grown crystals contain three times more needle crystallites of an impurity phase of PbI(OH)." (9) Notably, both the Earth and space crystals were similar in terms of defects relating to site vacancies and plane slippage. (9)

"In two experiments on Earth, with the reactors oriented so that their membrane templates stood vertically, yellow lead iodide crystals formed only on the lower half of each membrane. A deep, horizontal, lead iodide shelf marked the upper boundary of crystal growth. Crystalline supports flared out beneath it, and a beard that grew from the shelf began dripping crystals onto the growth chamber floor within 20 minutes.

"In contrast, a relatively even... [mound] of crystals smothered the entire surface of the template membranes in each of the three space-borne reactors. Additional satellite growths... crystallized throughout the adjacent liquid--a phenomenon that... [had] never been witnessed in Earth-grown crystals offered a template on which to adhere." (3, p. 206)

<Note: Not all of the references listed below were used to prepare this experiment summary.>

Key Words: Crystal Growth From Solution, Aqueous Solutions, Reactant Solutions, Insoluble Crystals, Single Crystals, Double Replacement Reaction, Ion Exchange, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Concentration Distribution, Semipermeable Membranes, Growth Rate, Solid/Liquid Interface, Liquid/Liquid Interface, Surface Morphology, Crystal Morphology, Sample Purity, Crystalline Defects, Needles, Hardness, Contamination Source, Impurities, Liquid Reservoir, Contained Fluids, Pictoral X-Ray Enhancement

Number of Samples: three, three-chambered growth reactors
Sample Materials: Solutions in the three chambers: (1) lead acetate $Pb(C_2H_3O_2)_2$, (2) potassium iodide (KI), (3) water.
Membrane Material: natural cellulose
(Pb*C*H*O*, K*I*)
Container Materials: acrylic Plexiglas™

Experiment/Material Applications:
"Purer lead iodide crystals could boost the sensitivity of X-ray and gamma-ray film--potentially allowing physicians to reduce patient X-ray exposures without sacrificing image quality.... The shuttle experiments also suggest space-grown crystals might yield significant improvements for "molecular sieves," a commercially important class of chemical filters made from tightly packaged crystals." (3, p. 206)

References/Applicable Publications:
(1) Report to Educators. NASA Publication, Vol. 16, Number 2, Volume 16, Number 3, Summer/Fall 1988, p. 6. (preflight)

(2) "Seven Marshall Payloads to Fly on STS-26 in June." Marshall Star, Volume 28, No. 5, October 7, 1987, pp. 1-2. (preflight; very short description)

(3) Raloff, J.: "Better Crystals? It's a Matter of Space," Science News, 1989, Vol. 136, p. 206. (post-flight)

(4) NASA Press Kit, Space Shuttle Mission: STS-26, September 1988, p. 36. (preflight)

(5) Haggin, J.: "Lead Iodide Crystals Studied in Space," Chemical and Engineering News, 1989, Vol. 67(39), p. 38.

(6) Van Pelt, D.: "Space-Grown Crystals Have Earthly Potential," Insight, October 23, 1989, p. 48.

(7) Scaife, C.W.J., Cavoli, S. R., and Suib, S. L.: "Crystallization in Space: Implications for Molecular Sieve Synthesis." In ACS Symposium Series 437, Novel Materials in Heterogeneous Catalysis, R.T.K. Baker and L. L. Murrell, editors, American Chemical Society, Washington, D.C., 1990, pp. 2-13.

(8) Scaife, C.W.J., Cavoli, S. R., Blanton, T. N., Morse, M. D., Sever, B. R., Willis, W. S., and Suib, S. L.: "Synthesis and Characterization of Lead(II) Iodide Grown in Space." Chemistry of Materials, 1990, 2, 777.

(9) Input received from Co-Investigator C. W. Scaife, June 13, 1991 and July 1993.

(10) Input received from Principal Investigator, S. R. Cavoli, July 1993.

(11) Crystal Growth Using a Semi-Permeable Membrane. In Shuttle Student Involvement Program (SSIP) Final Reports of Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989.

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Co-Investigator(s): Unknown
Affiliation(s): (1,2) College of Pharmacy, University of Arizona, Tucson; (3) Department of Chemical Engineering, University of Arizona, Tucson; (4) National Institute of Standards and Technology, Boulder, Colorado

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Phenytoin Precipitation

"The crystallization of pharmaceutical products, such as phenytoin ("Dilantin") is, to the extent possible, adjusted to optimize the absorption of the drug [into the body]. Nucleation and growth processes determine the final crystal size distribution and thus, can affect the absorption characteristics. It is thought that gravity may influence either or both of these steps." (5, p. 1)

Phenytoin is precipitated via a pH change: "When a solution of phenytoin dissolved at pH 11 is mixed with one of lower pH (say pH 2.0) the resulting solution will have an intermediate value of pH and, depending upon the pH 11 phenytoin concentration, can be supersaturated." (5, p. 1) In the low-gravity environment, "...contact between the container and the solution can be eliminated[.] This removes sites for heterogeneous nucleation and it may be possible to produce a solid precipitate via homogeneous nucleation." (1, Viewgraphs)

The objective of this phenytoin precipitation experiment was to study (1) the resultant crystal size distribution when gravity-dependent convective and sedimentary forces are reduced and (2) to nucleate uniform crystals of a pharmaceutical product.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Schoonen, Consort 1 (this

chapter); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1,2,11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments) the apparatus was also used to (1) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (2) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (1) aligned or (2) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto et al., Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted seven "Type 3" test wells. Discussion of this specific well-type is detailed here.

Each "Type 3" test well provided the investigator with one experimental opportunity. The well-type used three sample wells, two in the top block and one in the bottom block. One of these wells on the top block "...contained a .45μm pore size membrane filter backed and supported by a cellulose prefilter. The filters were prepared by cutting small circles from normal filters with a cork borer." (5, p. 1) The second well on the top block of each well-type contained the high pH solution (6.28 mg/g of phenytoin at pH 11). The well on the bottom block contained the low pH solution (pH 2.0 phosphate buffer).

Prior to the Consort flight, the blocks were joined together such that the wells in the upper block were purposely misaligned with the well in the lower block. Once the rocket had been launched

and the low-gravity period achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells containing the phenytoin solution and pH 2.0 buffer. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity phase, the upper block again moved right, aligning the well which now contained a mixture of liquid pH 2.0 buffer and the phenytoin crystals with the top well containing the filter. These wells remained in contact through the re-entry phase of the flight. Reportedly, "Filtration was expected to be accomplished by either acceleration forces moving the fluid through the filter or by capillary action drawing fluid through the filter into the absorbent backing used to support the filter." (5, p. 2)

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 3" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by this form of contamination.

Examination of the seven Consort 1 pH 2.0 phosphate buffer wells indicated that (1) filtration of the contents during re-entry had occurred, (2) no crystals were present in the remaining solution and (3) that the pH of the fluid in the seven wells ranged from 2.0 to 2.2.

Examination of the seven Consort 1 filter wells indicated that the filters worked adequately. Reportedly, (1) there were very few crystals in the filters and (2) each of the filters exhibited some contamination from a silicon grease source.

Examination of the seven Consort 1 phenytoin solution wells indicated that the pH in the wells ranged from 6.0 to 10.4. Such a variation in pH was not expected. "During the entire flight, the solution was either isolated from all other wells or in contact with the ph [sic] 2.0 solution in the lower wells. Mixing between the lower and upper wells was presumed to be by diffusion only. Since all seven wells contained identical solutions, identical diffusion mixing should have occurred in each....

"This is not supported by the estimates of mixing required to produce a given pH...." (5, p. 8)

It was reported that a large number of crystals were present in five of the wells and no crystals were present in the remaining two wells. The two wells which did not contain crystals had a pH of 10.2 or higher. In four of the five wells which had crystals, the usual phenytoin habit of long needle-shaped crystals was ob-

served and there was no wide dispersion of crystal sizes. The fifth well, however, "...contained crystals markedly different than those observed in the laboratory. The distribution consisted of one or two very large needle shaped crystals and numerous small, rounded crystals.... Closer examination of the large needle shaped crystals showed a very rough surface with large pits, steps and cracks. It is hypothesized that the small crystals were formed from breakage or chipping from the large crystals..." (5, p. 3) possibly during payload re-entry. Further, it was reported that some of these wells experienced an unexplained 25%-50% fluid loss. "Because... [the wells] were either isolated from all other wells or in contact with only the bottom wells containing pH 2.0 buffer, there should have been no fluid loss from them." (5, p. 8)

Other assessments of the low-gravity formed crystals were not reported at this time.

The rocket samples were compared to ground-based samples which had been similarly processed in the MDA (1) with the pH 11 phenytoin solution on the bottom and the pH 2 solution on the top and (2) with the pH 2 solution on the bottom and the pH 11 phenytoin solution on the top.

Examination of the ground-based pH 2 solution wells (top) indicated that (1) crystals were found in one-third of the wells, (2) the pH in the wells ranged from 2.0 to 5.8, and (3) the crystals were formed in the wells with high pH values (5.6 and 5.8). Examination of the ground-based pH 11 phenytoin solution wells (bottom) indicated that (1) crystals were found in two-thirds of the wells and (2) the pH in all of the wells ranged from 2.4 to 10.4. "Unlike the microgravity results, copious crystal precipitation was seen in... [1/3 of the] wells that originally contained the pH 2.0 phosphate buffer solution. However, the crystal appearance was similar to that of the crystals found in wells containing the pH 11 phenytoin solution. There was no apparent change in the number of crystals produced or in their size." (5, p. 9) A significant change in pH occurred in the sample wells. For some wells the variation in pH was larger than in the low-gravity Consort experiments. The variation difference was attributed to (1) gravity-independent effects (such as diffusion) and (2) gravity-dependent effects (such as convection), both of which dictated the mixing of the solutions in the upper and lower wells.

Examination of the ground-based pH 2 solution wells (bottom) indicated that (1) no crystals were found in any of the wells and (2) the pH in all the wells ranged from 2.1 to 2.4. Examination of the ground-based phenytoin solution wells (top) indicated that (1) no crystals were found in any of the wells and (2) the pH

ranged from 10.3 to 10.6. "The lack of crystalline product... was not due to the presence of gravity. Mixing of the solutions used in... [this] experiment on a microscope slide [presumably at some later date] produced immediate precipitation of crystals with similar appearance, size and numbers to those produced in [the other ground-based experiment]." (5, p. 9) Little pH change occurred in the wells during this ground-based experiment. "While the wells were prepared in the same manner as those in... [the other ground-based experiment] and filled to the same levels, apparently little or not contact was made between any of the top and bottom wells." (5, p. 9)

Very little fluid loss was observed in either of the ground-based experiments except in one of the wells.

Key Words: Crystal Growth From Solution, Biotechnology, Pharmaceutical Products, Pharmaceutical Applications, Model Materials, pH Level, Precipitation, Supersaturation, Buoyancy-Driven Convection, Sedimentation, Diffusion, Diffusive Mass Transfer, Liquid/Liquid Diffusion, Double Diffusion, Filtration, Liquid/Liquid Interface, Membranes, Capillary Flow, Capillary Forces, Nucleation, Heterogeneous Nucleation, Nucleation Sites, Solid/Liquid Interface, Surface Morphology, Cracks, Needles, Contained Fluids, Crucible Effects, Liquid Leakage, Contamination Source, Acceleration Effects, Vehicle Re-entry Forces/Vibration, Payload Survivability

Number of Samples seven
Sample Materials: Top wells: (1) high pH solution of (6.28 mg/g of phenytoin at pH 11), (2) a 0.45 μm pore size membrane filter backed and supported by a cellulose prefilter/air; bottom well: pH 2.0 phosphate buffer
Container Materials: unclear, possibly inert material

Experiment/Material Applications:
"Phenytoin is being studied as a model compound for organic electrolytes. Elucidation of the mechanisms of precipitation and growth will provide a basis for future improvements in drug dosage design." (1, Viewgraphs)

"With an understanding of nucleation and growth mechanisms a drug can be produced with an optimum size distribution for absorption into the body." (1, Viewgraphs)

References/Applicable Publications:

(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter, January 1991)

(5) Phenytoin Crystallization in Microgravity. Final Report of the Consort I Flight Results, November 14, 1989, In: Correspondence from Gail M. Zipp (University of Michigan) to Dr. Paul Todd (National Institute if Standards and Technology), November 14, 1989. (provided by ITA to C. A. Winter, January 1991; post-flight)

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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Inorganic Crystallization

"Formation of crystalline phases from solution is affected by [the] Earth's gravitational field. The... field induces [(1)] settling of particles and [(2)] convection at growing crystal faces. Settling causes particles to agglomerate, leading to a wide range in particle size; convection at a growing crystal face is one of the processes responsible for crystal defects." (1, p. 1)

On Earth, "...dispersions of micro-crystalline material are only stable if the thermal motion of the particles (Brownian Motion) counterbalances the gravitational force exerted on the particles. However, both Brownian motion and the gravitational field cause particles to agglomerate, eventually leading to [a] particle too big to stay in dispersion.... To prevent agglomeration, the surface of the particles is charged, which generates a coulombic repulsion between approaching particles.... Preparing a dispersion of negatively charged silver iodide particles is very easy, because the surface is capable of adsorbing excess iodide anions." (1, p. 1)

The objective of this Consort 1 experiment was to study how the absence of settling in the low-gravity environment affects the particle size distribution of microcrystalline dispersions.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chpater 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (this chapter); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1,2,11); Vera, Consort 1 (two experiments,

Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments (including this one)), the apparatus was also used to (1) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (2) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (1) aligned or (2) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto et al., Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted one "Type 2" test well. Discussion of this specific well-type is detailed here.

The single "Type 2" test well provided the investigator with one experimental opportunity. The well-type used two sample wells, one on the top block and one on the bottom block. Prior to flight, each well was filled. The top well was filled with sodium iodide (0.1 M concentration) and the bottom well was filled with $AgNO_3$ (0.1 M concentration).

Prior to rocket launch, the blocks were joined together such that the well in the upper block was purposely misaligned with the well on the lower block. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity rocket phase, the upper block again moved right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the

liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

A document written prior to the launch of the Consort 1 rocket detailed the expected post-flight analysis and results:

"...the size distributions of a negatively charged and an uncharged silver iodide dispersion will be compared to similar dispersions prepared under the influence of Earth's gravitational field." (1, p. 1)

"Dispersions of charged particles are expected to show a very narrow size distribution, irrespective of the gravitational field. However, the size distribution of uncharged dispersions grown in zero-gravity is expected to be markedly different from uncharged dispersions grown under the influence of the Earth's gravitational field. Any spread in size distribution of an uncharged dispersion grown in zero-gravity is solely due to Brownian motion, whereas the size distribution of an uncharged dispersion grown under the influences of gravity is due to <u>both</u> gravity and Brownian motion." (1, p. 1-2)

Reference (2) indicated that as of 4/14/89, the experimental results were still being analyzed; Reference (1) indicated that the post-flight analysis of the 200 μl dispersion would be examined using a Scanning Transmission Electron Microscope (STEM). The STEM was expected to allow (1) the determination of the particle size and (2) an assessment of the crystalline morphology of individual particles.

It was reported that "We encountered serious problems in imaging the AgI crystals with STEM. While focusing the beam the crystals would deform. This made analysis impossible. However, it was clear that AgI did form." (6)

No other information concerning this experiment could be located at this time.

Key Words: Crystal Growth From Solution, Inorganic Crystallization, Sedimentation, Buoyancy-Driven Convection, Diffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Brownian Motion, Liquid/Liquid Interface, Solid/Liquid Interface, Charged Particles, Particle Dispersion, Particle Size Distribution, Particle Agglomeration, Microcrystalline Dispersion, Crystalline Defects, Crystal Morphology, Liquid Leakage, Contamination Source, Deterioration of Samples After Low-G Flight

Number of Samples: one
Sample Materials: Top well: sodium iodide (0.01 M concentration); bottom well: $AgNO_3$ (0.1 M concentration)
(Ag*N*O*, Na*I*)
Container Materials: Appears to have been an inert material.

Experiment/Material Applications:
A brief note in Reference (2) indicated that this investigation had commercial applications in the area of materials research. No further details concerning the research applications were explicitly cited in the available references. However, it seems reasonable to assume that if particles did not settle in the low-gravity environment, the particle size distribution of microcrystalline dispersions could be better controlled and crystalline defects could be reduced.

References/Applicable Publications:
(1) Schoonen, M.A.A. and Reeder, R. J.: Proposal to Study Dispersions of Inorganic Microcrystalline Phases Grown in Zero Gravity. Information sent by ITA, April 3, 1990, to C. Winter (NASA). (preflight)

(2) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (postflight)

(3) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(4) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results- A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(5) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter, January, 1991)

(6) Input received from Principal Investigator M. Schoonen, August 1993.

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CHAPTER 9

CRYSTAL GROWTH FROM THE MELT

Principal Investigator(s): Witt, A. F. (1), Gatos, H. C. (2)
Co-Investigator(s): Unknown
Affiliation(s): (1,2) Massachusetts Institute of Technology, Cambridge, Massachusetts

Experiment Origin: USA
Mission: Apollo. Although this experiment was originally planned for the early Apollo space program, the investigation was eventually adopted for the Skylab program (see Witt, Skylab SL-3).
Launch Date/Expt Date: Not applicable
Launched From: Not applicable
Payload Type: Planned: Apollo Command Module Payload
Processing Facility: Planned facility: unknown
Builder of Processing Facility: Unknown

Experiment:
Apollo Indium Antimonide Remelt

This Apollo experiment was the first in a series of investigations designed by Witt et al. to study low-gravity crystal growth from the melt.

Samples of indium antimonide, doped with tellurium, were prepared for processing under low-gravity conditions during an early Apollo mission. <Note: It is not clear which Apollo mission was to carry the experiment.>

Reportedly, "Although all crystals were ready for the Apollo mission, the experiment was not finally realized. At the recommendation of the National Aeronautics and Space Administration all materials and techniques developed were adopted for the... [Skylab] experiment of growing indium antimonide from the melt under zero gravity conditions." (1, p. 2)

No further information concerning this experiment could be located.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Ternary Systems, Dopant, Electronic Materials, Sample Not Processed As Planned

Number of Samples: It is unclear how many samples were originally planned.

Sample Materials: indium-antimony doped with tellurium (In*Sb*, Te*)
Container Materials: It is unclear which material was originally selected.

Experiment/Material Applications:
See Witt, Skylab, SL-3.

References/Applicable Publications:
(1) Gatos, H. C. and Witt, A. F.: Apollo Indium Antimonide Remelt Experiment. Final Report, NASA CR-149996, October 1972, 2 pp.

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Co-Investigator(s): Unknown
Affiliation(s): (1,2) Massachusetts Institute of Technology, Cambridge, Massachusetts

Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt Date: September 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) Area; Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Indium Antimonide Crystals (M562)

This Skylab SL-3 experiment was the second in a series of investigations designed by Witt et al. to study low-gravity crystal growth from the melt (see Witt, Apollo). The specific objectives of the experiment were to (1) confirm the advantages of low-gravity solidification, (2) obtain basic data on solidification, and (3) investigate the feasibility of processing electronic materials in a reduced-gravity environment. It was expected that the experiment would (1) demonstrate diffusion-controlled, steady state solidification and (2) allow investigation of the micro-and macro-segregation behavior of InSb processed in space.

Prior to the Skylab SL-3 mission, two sets of samples were prepared. One of the sets was used during the Skylab SL-3 mission; the other set was intended as a backup. <Note: the backup samples were eventually used for the second Skylab experiment by Witt (see Witt, Skylab SL-4 (this chapter)).> Each sample set consisted of (1) one undoped InSb sample, (2) one Te-doped InSb sample, and (3) one heavily Sn-doped InSb sample. The single crystal samples were prepared on Earth using the Czochralski technique (<111> direction). After grinding each to a diameter of 1.4 cm and a length of 17 cm, the crystals were chemically etched to remove surface damage and further reduce the sample diameter to the desired value. Each crystal was placed into a quartz ampoule (wall thickness of 3 mm) with graphite spacers at the sample ends. (The diameter of each of the crystals was 0.25 mm smaller than the diameter of the ampoules.) The crystals were loaded such that the B<111> direction coincided with the regrowth direction. Prior to sealing, the ampoules were repeatedly flushed with helium and then evacuated to 10^{-7}

Torr. Each ampoule was sealed in an evacuated stainless steel cartridge.

The experiment was performed in the M518 Multipurpose Electric Furnace System. The apparatus consisted of three main sections: (1) the furnace (which interfaced with the Skylab M512 materials processing facility), (2) a programmable electronic temperature controller, and (3) the experiment cartridges. The furnace contained three cavities, which accommodated the three prepared stainless steel cartridges. Three separate heat zones were maintained in each cavity: (1) a constant temperature hot zone (up to 1000 ^{o}C), (2) a gradient zone (20 to 100 ^{o}C/cm), and (3) a cool zone. (In the cool zone, heat was conducted by radiation.)

During the mission, melting was initiated at one end of the crystal (hot end), and the solid-liquid interface was advanced to the desired position (back-melting) by the appropriate power input. (Back-melting was accomplished after heating for approximately 120 minutes.) The samples were then held at temperature for 60 minutes (soak period) to achieve sample isothermality and homogenization. Regrowth of the crystal (accomplished via cooling at a rate of 1.17 ^{o}C/cm) was achieved by controlled power reduction. Four hours after initiation of regrowth, the power was turned off and the samples were passively cooled to ambient. (See Reference (1) for thermal history details.)

Post-flight, it was reported that the surfaces of the undoped and Sn-doped InSb samples were "...smooth and highly reflective, indicating that they formed in intimate contact with the confining walls. The presence of randomly distributed cavities of varying size, attributed to entrapped gas, indicates that the melt was also in intimate contact with (wetted) the quartz walls. The morphological characteristics of the two crystals are identical; differences in phase (Sn) segregation are associated with differences in growth conditions." (1, p. 278) <Note: No other results concerning these samples were reported.>

Examination of the Te-doped crystal indicated that the sample grew without contact with the ampoule wall. The surface of the crystal was dull in appearance and did not contain peripheral cavities. External boundaries of rotational twins, exhibited as several bands of varying width, were located on the surface of the regrown crystal in the vicinity of the seed. Also present on the crystal surface were ridges, which were shiny at the top indicating contact with the container wall. On average, they were 25 microns high and increased in width toward the hot end of the sample. Over the last 10 mm of the sample, the ridges became irregular and branched out. The areas between the ridges generally exhibited characteristics of free surface growth.

<Note: The conclusions from the Skylab SL-3 experiment were reported with those from a subsequent investigation conducted during the Skylab SL-4 mission. Therefore, these conclusions are presented under Witt, Skylab SL-4.>

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Steady-State Solidification, Thermal Gradient, Diffusion, Diffusion-Controlled Growth, Dopant, Binary Systems, Ternary Systems, Electronic Materials, Surface Morphology, Segregation, Macrosegregation, Microsegregation, Phase Segregation, Single Crystals, Seed Crystals, Wetting, Wetting of Container, Non-Wetting of Container, Crucible Effects, Free Surface, Sample Detachment from Crucible, Free Surface Solidification, Solid/Liquid Interface, Radiative Cooling, Passive Cooling, Cooling Rate, Thermal Soak, Sample Homogeneity, Cavity, Gas Formation, Rotational Twinning, Vacuum

Number of Samples: three
Sample Materials: (1) undoped InSb, (2) tin-doped (approx. $10^{20}/cm^3$) InSb, and (3) tellurium-doped (approx. $10^{18}/cm^3$) InSb (In*Sb*, In*Sb*Sn*, In*Sb*Te*)
Container Materials: Quartz ampoules held in stainless steel cartridges.
(Si*O*)

Experiment/Material Applications:
Indium antimonide was selected as the experimental material because (1) its relatively low melting point (525 ^{o}C) was compatible with the furnace hardware capabilities and (2) chemical etching, which was the only high-resolution technique available for microsegregation studies, was well developed for InSb.

References/Applicable Publications:
(1) Witt, A. F., Gatos, H. C., Lichtensteiger, M., Lavine, M. C., and Herman, C. J.: Steady State Growth and Segregation Under Zero Gravity: InSb. In NASA Marshall Space Flight Center Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. 1, June 1974, pp. 275-299. (post-flight)

(2) Witt, A. F., Gatos, H. C., Lichtensteiger, M., Lavine, M. C., and Herman, C. J.: Crystal Growth and Steady-State Segregation Under Zero Gravity: InSb. Journal of the Electrochemical Society, Vol. 122, No. 2, February 1975, pp. 276-283.

(3) Naumann, R. J. and Herring, H. W.: Experiment M562, Steady State Growth and Segregation Under Zero Gravity of Indium Antimonide. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, pp. 54-55. (post-flight results)

(4) Experiment M562-Indium Antimonide Crystals. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-72 - 5-74. (post-flight)

(5) M518-Multipurpose Electric Furnace Experiments. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(6) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(7) Multipurpose Electric Furnace (M518). MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(8) Indium Antimonide Crystals (M562). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-51. (post-flight; very short summary)

(9) Bourgeois, S. V., Jr. and Spradley, L. W.: Thermocapillary Convection in Microgravity Crystal Growth Melts of Indium-Antimonide. Letters in Heat Mass Transfer, Vol. 3, 1976, pp. 193-204. (related research)

(10) Wiedemeier, H.: Crystal Growth in Micro-Gravity - An Overview. In Applications of Space Flight in Materials Science and Technology, proceedings of a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977 (issued September 1978), pp. 25-39. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt Date: January 1974 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area; Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Indium Antimonide Crystals (M562)

This Skylab SL-4 experiment was the third in a series of investigations designed by Witt et al. to study low-gravity crystal growth from the melt (see Witt, Apollo, Skylab SL-3). The specific objectives of the experiment were to (1) confirm the advantages of low-gravity solidification, (2) obtain basic data on solidification, and (3) investigate the feasibility of low-gravity processing of electronic materials. It was expected that the experiment would (1) demonstrate diffusion-controlled, steady state solidification and (2) allow investigation of the micro- and macro-segregation behavior of InSb processed in space.

The Skylab SL-3 backup sample set was used for this SL-4 mission. The set consisted of (1) one undoped InSb sample, (2) one Te-doped InSb sample, and (3) one heavily Sn-doped InSb sample. (The preflight sample preparation procedures are detailed under Witt, SL-3.)

The experiment was performed in the M518 Multipurpose Electric Furnace System. The apparatus consisted of three main sections: (1) the furnace (which interfaced with the Skylab M512 materials processing facility), (2) a programmable electronic temperature controller, and (3) the experiment cartridges. The furnace contained three cavities, which accommodated the three prepared stainless steel cartridges. Three separate heat zones were maintained in each cavity: (1) a constant temperature hot zone (up to 1000 ^{o}C), (2) a gradient zone (20 to 100 ^{o}C/cm), and (3) a cool zone. (In the cool zone, heat was conducted by radiation.)

During the mission, melting was initiated at one end of the crystal (hot end), and the solid-liquid interface was advanced to the desired position (back-melting) by the appropriate power input. (Back-melting was accomplished after heating for approximately 120 minutes.) The samples were then held at temperature for 60 minutes (soak period) to achieve sample isothermality and homogenization. Regrowth of the crystal (accomplished via cooling at a rate of 1.17 ^{o}C/cm) was achieved by controlled power reduction. The samples were subjected to mechanical shock by striking the furnace assembly at a predetermined time. Also, after initiation of regrowth, the SL-4 samples were subjected to a second thermal soak period of 60 minutes. These two changes in the experiment procedure (over the SL-3 experiments) "...were intended to provide time reference markings in the crystal and to obtain data on the dependence of transient segregation on growth rate." (1, p. 278)

Post-flight, it was reported that, for both Skylab SL-3 and Skylab SL-4 mission samples, the surfaces of the undoped and Sn-doped InSb samples were "...smooth and highly reflective, indicating that they formed in intimate contact with the confining walls. The presence of randomly distributed cavities of varying size, attributed to entrapped gas, indicates that the melt was also in intimate contact with (wetted) the quartz walls. The morphological characteristics of the two crystals are identical; differences in phase (Sn) segregation are associated with differences in growth conditions." (1, p. 278) <Note: No other results concerning these samples were reported.>

Examination of the Te-doped crystal grown during the Skylab SL-4 mission indicated that the sample regrew with first a decreasing diameter followed by nearly a constant diameter. With continued growth, the diameter again decreased and later increased in size. Eventually, the diameter assumed a constant size of 12.8 mm, which was the same as the i.d. of the quartz ampoule. "From volume considerations, it is concluded that the increase of the crystal diameter, with continuing growth coincides in time with the initial contact of the melt with the graphite spacer at the end of the quartz ampoule. Since crystal growth over the first 30 mm proceeded with a decreased diameter, and solidification of InSb is accompanied by a volume expansion of 12.9%, the volume available to the residual melt is less than during the Skylab-III experiment at the same time in growth. Thus, towards the final stages of solidification some melt was ultimately forced into the peripheral cavity available in the hot-end graphite spacer.... Since the clearance between the spacer and the quartz wall was less than 0.1 mm, the melt was under substantially increased pressure during the last 25 mm of growth. This increased pressure, however, did not lead to forced wetting between the melt and the quartz wall since contact between the crystal and the

wall remained confined to the surface of the irregularly spaced ridges as in the Skylab-III crystal." (1, p. 280)

Using various analytical techniques, the bulk characterization of the Skylab SL-3 and Skylab SL-4 samples led to the following results and conclusions:

(1) Ideal steady state growth and segregation was achieved during low-gravity processing. This diffusion controlled growth resulted in a "...three-dimensional chemical homogeneity on a microscale over macro-scale dimensions (several centimeters in the present case)...." (1, p. 287) Characterization of the transient segregation profile preceding the steady state was also accomplished. However, limitations in microanalytical techniques (available at the time) did not allow determination of the fundamental solidification data.

(2) The Te-doped InSb samples (Skylab SL-4) solidified with a free surface due to surface tension effects. Reportedly, this phenomenon had not been previously observed. It was reported that these surface tension effects remained localized on the surface and did not affect bulk growth or segregation. It would be expected that this free surface condition would lead to a surface tension-driven Marangoni flow within the melt. However, no such flows were detected. It was reported in Reference (3) that the lack of fluid flow may have been due to the presence of a thin oxide film on the free surface.

(3) Without the interference of convective flows, it was possible to identify segregation discontinuities caused by faceted growth and explain their presence on the basis of spurious nucleation. The absence of convection also allowed "...the determination of the mode of nucleation (formation of misoriented nuclei at the three phase boundary line) and propagation of rotational twinning." (1, p. 287)

(4) The perturbation caused by mechanical shock to the system (Skylab SL-4 samples) caused a localized increase in dopant segregation. This discontinuity allowed the determination of the average macroscopic growth rate. The average growth rate from the initial regrowth to the application of mechanical shock (90 minutes into regrowth) was 2.8 microns/sec. From this point to the second discontinuity, the growth rate was 4.6 microns/sec. Over the first 2.88 cm of regrowth, the average growth rate was 3.4 microns/sec.

It was finally concluded that low-gravity experimentation can provide the fundamental data required for bridging the gap between theory and experiment in the area of solidification.

<Note: Further details concerning the results and conclusions from these experiments can be located in Reference (1) and Reference (2).>

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Steady-State Solidification, Thermal Gradient, Diffusion, Diffusion-Controlled Growth, Dopant, Binary Systems, Ternary Systems, Electronic Materials, Surface Morphology, Segregation, Macrosegregation, Microsegregation, Phase Segregation, Wetting, Free Surface, Free Surface Solidification, Surface Tension, Solid/Liquid Interface, Radiative Cooling, Thermal Soak, Interface Demarcation, Sample Homogeneity, Cavity, Gas Formation, Rotational Twinning, Facets, Growth Rate, Spurious Nucleation, Sample Deformation, Volume Change, Volume Expansion, Sample Shrinkage, Sample Detachment from Crucible, Non-Wetting of Container, Liquid Transfer, Thin Films, Oxide Layer, Contamination Source, Coated Surfaces

Number of Samples: three
Sample Materials: (1) undoped InSb, (2) tin-doped InSb, and (3) tellurium-doped InSb
(In*Sb*, In*Sb*Sn*, In*Sb*Te*)
Container Materials: Quartz ampoules held in stainless steel cartridges.

Experiment/Material Applications:
See Witt, Skylab, SL-3.

References/Applicable Publications:
(1) Witt, A. F., Gatos, H. C., Lichtensteiger, M., Lavine, M. C., and Herman, C. J.: Steady State Growth and Segregation Under Zero Gravity: InSb. In NASA Marshall Space Flight Center Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. 1, June 1974, pp. 275-299. (post-flight)

(2) Witt, A. F., Gatos, H. C., Lichtensteiger, M., Lavine, M. C., and Herman, C. J.: Crystal Growth and Steady-State Segregation Under Zero Gravity: InSb. Journal of the Electrochemical Society, Vol. 122, No. 2, February 1975.

(3) Naumann, R. J. and Herring, H. W.: Experiment M562, Steady State Growth and Segregation Under Zero Gravity of Indium Antimonide. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, pp. 54-55. (post-flight results)

(4) Experiment M562-Indium Antimonide Crystals. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-72 - 5-74. (post-flight)

(5) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(6) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(7) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop. NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(8) Indium Antimonide Crystals (M562). In MSFC Skylab Mission Report-Saturn Workshop. NASA TM X-64814, October 1974, p. 12-51. (post-flight; very short summary)

(9) Bourgeois, S. V., Jr. and Spradley, L. W.: Thermocapillary Convection in Microgravity Crystal Growth Melts of Indium-Antimonide. Letters in Heat and Mass Transfer, Vol. 3, 1976, pp. 193-204. (related research)

(10) Wiedemeier, H.: Crystal Growth in Micro-Gravity - An Overview. In Applications of Space Flight in Materials Science and Technology, proceedings of a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977 (issued September 1978), pp. 25-39. (post-flight)

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Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Docking Module Payload
Processing Facility: Multipurpose Electric Furnace (MPEF) gradient furnace located in ASTP docking module: the Multipurpose Furnace of Skylab, "...modified for ASTP to meet the higher temperature requirements." (2, p. 430)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Interface Marking in Crystals (MA-060)

This ASTP experiment was the fourth in a series of investigations designed by Witt et al. to study low-gravity crystal growth from the melt (see Witt, Apollo, Skylab SL-3, Skylab SL-4).

The earlier Skylab experiments by Witt which had examined the crystal growth of indium antimonide, established that the low-gravity environment can provide virtually ideal conditions for solidification studies. Thus, the ASTP experiment was designed to obtain more refined fundamental and quantitative information on the solidification and segregation processes. The specific objectives of the experiment were to examine the following low-gravity processing characteristics of a germanium doped gallium system:

(1) the absence or presence of convective phenomena

(2) the surface tension and/or wetting of the molten material

(3) the microscopic growth rate behavior during directional solidification (and the corresponding segregation)

(4) the heat transfer of the experimental system

Prior to the mission, three germanium (Ge) crystals doped with gallium were prepared. One Ge crystal was grown in the <100> direction; another was grown in the <111> direction. <Note: The growth direction of the third crystal could not be determined from the published literature.> Each sample was sealed in its

own evacuated quartz ampoule and placed within a stainless steel cartridge.

The experiments were performed in the three-chambered Multipurpose Electric Furnace (MPEF). This furnace, which had been used during the previous Skylab experiments by Witt, had been modified to meet the higher temperature requirements of the ASTP experiment (see Witt, Skylab SL-3, for additional information).

During the ASTP mission, the three samples were simultaneously melted in the furnace by applying power to the heating elements. The solid-liquid interface was established after 6 cm of each crystal was melted. After thermal soaking for 2 hours, the power system was switched to a cool-down mode (resulting in an average cooling rate of 2.4 ^{o}C/min). This cooling rate was expected to yield a microscopic growth rate of 5 microns/sec initially and 10 microns/sec at the end of the cool-down period. Controlled cool-down was halted after 83 minutes (approximately 4 cm regrowth length) at which time passive cool-down was allowed for 67 minutes. After this period, helium was injected into the furnace to accelerate cooling.

At 4-second intervals, throughout the experiment, the samples were subjected to current pulsing that allowed demarcation of the solid-liquid interface. To accomplish this procedure, graphite cup electrodes with platinum leads were attached to the bottom and top of each sample. The platinum leads of the three samples were connected in series with the seed material having a positive polarity. The current pulses were 19.1 A/cm^2 with a duration of 55 milliseconds. "The interface demarcation technique was incorporated into the experiment because it constitutes a unique tool for recording the morphology of the growth interface into the grown crystal, for determining the microscopic growth rate throughout solidification, and for establishing an absolute time-reference framework for all stages of the solidification process." (2, p. 429)

Post-flight, it was reported that all space-grown crystals could be readily removed from their quartz ampoules, while those processed on Earth required removal by chemical dissolution of the quartz. The space crystals also exhibited the network of ridges seen on the Skylab InSb specimens (see Witt, Skylab SL-3). "It was therefore concluded that no wetting contact existed between the Ge melts and the confining quartz ampoules during growth in space; whereas, during growth on Earth, the Ge melt was in wetting contact with its confinement. This conclusion is significant because it suggests that effective wetting inversion... is a phenomenon that may be expected to occur in a multitude of systems subjected to solidification in space." (2, p. 431)

Examination of the morphological characteristics (e.g., absence of twin and grain boundaries) revealed that the Ge <111> sample maintained its single crystallinity in the peripheral region. This was not the case for the <111> sample grown on Earth. However, the space-processed Ge <100> sample exhibited surface grain boundaries after the first 3.5 cm of regrowth. The same sample processed on Earth exhibited grain boundaries after 2 cm of growth.

High-resolution characterization of the dopant distribution indicated that segregation occurred after the initial growth transient (unlike the previous Skylab experiments). Dopant concentration increased steadily over the first 1.5 cm of regrowth, but never achieved the steady-state prediction. It was also determined that the distribution of dopant varied with distance from the axis of the crystal. It was concluded, through analysis of the interface markings, that these results were probably caused by asymmetrical heat flow through the furnace. Therefore, flat solid-liquid interfaces were not present during the experiment causing unequal growth rates and radial segregation. The asymmetrical heat flow was probably present during the Skylab experiments but was not as pronounced due to (1) the difference in segregation coefficient of the materials used during Skylab and (2) the higher resolution techniques used for the ASTP samples.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Seed Crystals, Single Crystals, Steady-State Solidification, Dopant, Dopant Distribution, Binary Systems, Electronic Materials, Surface Morphology, Segregation, Wetting, Non-Wetting of Container, Surface Tension, Free Surface, Free Surface Solidification, Sample Detachment from Crucible, Solid/Liquid Interface, Thermal Soak, Asymmetric Temperature Field, Interface Demarcation, Electrodes, Electric Field, Passive Cooling, Quench Process, Sample Homogeneity, Growth Rate, Heat Transfer, Grain Boundaries, Twin Boundary, Vacuum

Number of Samples: three
Sample Materials: gallium-doped germanium (seed concentration of 10.18×10^{18} atoms/cm^3)
(Ga*Ge*)
Container Materials: Quartz ampoules contained in stainless steel cartridges.
(Si*O*)

Experiment/Material Applications:
The Ge-Ga doped system was selected for this experiment because (1) it had been extensively studied on Earth and (2) it lends itself to detailed macro- and microscopic characterization.

References/Applicable Publications:
(1) Witt, A. F.: Crystal Growth and Segregation in Space: A Critical Assessment Based on Results Obtained During the ASTP Mission. In Space Research XIX, Proceedings of the Open Meetings of the Working Groups on Physical Sciences, Innsbruck, Austria, May 29-June 10, 1978, Oxford Pergamon Press, Ltd., 1979, pp. 503-506. (post-flight)

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(3) Boese, A., McHugh, J., and Scidensticker, R.: Multipurpose Electric Furnace. In Apollo-Soyuz Test Project, Summary Science Report, NASA SP-412, Vol. I, pp. 353-365. (post-flight)

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Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt Date: September 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area; Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Mixed III-V Crystal Growth (M563); Directional Solidification of InSb-GaSb Alloy Semiconductors

<Note: W. R. Wilcox conducted two crystal growth experiments during the Skylab program; one on SL-3 and one on SL-4. Because applicable references combined the results of these experiments, both the SL-3 and SL-4 summaries are combined here. The objectives, setup, and experimental procedures were identical for both experiments (except where noted).>

The SL-3 and SL-4 experiments were designed by Wilcox et al. to study the low-gravity solidification of semiconductor materials. The specific objective of the investigations was to determine if space processing methods could be used to grow high-quality, homogeneous bulk crystals.

Prior to each flight, three $In_xGa_{1-x}Sb$ ($x = 0.5$, 0.3, and 0.1) samples were prepared by (1) mixing the elements in a carbon-coated silica ampoule, (2) heating the ampoule to 900 ^{o}C, and (3) cooling the ampoule in air (see Reference (1) for details). Each of the sample ingots (9 mm in length) was then placed in a new carbon coated, 8 mm i.d. silica tube with graphite plugs and quartz wool springs. The tubes were backfilled to 10 Torr with helium and sealed. Each tube was placed in a stainless steel cartridge under a vacuum of less than or equal to 10^{-4} Torr.

During each Skylab mission, the three samples were processed simultaneously using a gradient freeze furnace. The furnace was configured with a radiation resistance heater, an insulated region, and cooling fins. The heater was powered-up to (1) 960 ^{o}C during the Skylab 3 experiment and (2) 1020 ^{o}C during the Skylab 4 experiment. The heater remained at this temperature for 16 hours, resulting in meltback of about half the samples. The heater was programmed to cool at a rate of 0.6 ^{o}C/min. This process initially avoided constitutional supercooling. However, as the heater cooled, the temperature gradient decreased and the freezing rate increased. As a result, constitutional supercooling eventually occurred in all samples. Samples of the same compositions were processed horizontally and vertically in the same furnace (with heater on top) under 1-g conditions.

Post-flight examination of the Skylab 3 samples revealed "...a wavy surface with a diameter slightly less that that of the ampoule." (4, p. 187) This was attributed to a lack of wetting of the ampoule by the melt. The 1-g, horizontally processed samples contained a depression along the top side of the sample caused by gravity.

The Skylab 4 samples and 1-g, vertically processed samples had diameters which indicated complete filling of the ampoules by the melt. The last solidified portions of all the samples contained a dendritic structure similar to the originally cast material. The directionally solidified portions of all the samples contained elongated, irregular grains, many with (111) twin boundaries.

"In the first analysis of the results it was erroneously assumed that curved boundaries were all grain boundaries while straight boundaries were (111) twins [1,4,7]. Subsequently [3,6] it was shown that 'the curved boundaries initially believed to delineate grains of random relative orientation are actually twin boundaries consisting of short segments that frequently change <111> directions during growth. The higher twin content in earth-processed ingots come about because new twins were frequently generated during solidification on earth, but very seldom during space processing." (12)

For all samples, the number of microcracks increased along the length of the sample, reportedly due to the increase in compositional inhomogeneities. The microcrack number also increased with InSb content. There did not appear to be a gravitational influence on the number of microcracks. Voids present in the low-gravity processed ingots were more uniformly distributed than in the 1-g processed samples. The void volume was lower in the 1-g, vertically-processed and Skylab 4 samples than in the 1-g, horizontally-processed and Skylab 3 ingots. "In the

horizontally-processed ingots there was a tendency for the voids to lie near the side of the ingot that was on top during processing, i.e., the melt-vapor surface." (4, p. 191)

It was speculated that the differences in defect density between the 1-g processed samples and low-gravity processed samples were caused by foreign particles. "It is believed that particles at a growing interface can cause nucleation of gas bubbles... twins... and grain boundaries. On earth the particles would tend to settle on the interface, and to repeatedly impact with the interface when carried by a convective stream in the melt. In space, on the other hand, some particles would remain dispersed in the melt, and would contact the interface only when the interface reached them. Thus, more twins would be formed on earth by small particles interacting with the interface more frequently." (4, p. 191)

Atomic absorption analysis was used to analyze the average Ga and In concentration of every third or fourth cross-sectional slice of the ingots (see Reference (1) for method). It was determined that the vertically-processed and Skylab samples had nearly identical concentration profiles while the horizontally-processed samples were quite different because of free-convective stirring present during processing. There were also changes in concentration in the originally cast sections of the ingot which "...must have occurred during the soak period prior to programming down the heater. Since the castings were not homogeneous, the solid-liquid interface would actually have be a two-phase 'mushy zone' in which recrystallization and compositional redistribution would be fairly rapid." (1, p. 352)

Electron microprobe analysis was performed at one millimeter intervals along one-inch lengths of the samples (see Reference (1) for sample preparation details and methods). Scatter in the mole fraction of InSb began to occur after several mm of resolidification in the vertically and space-processed samples. This result indicated a non-planar interface caused by constitutional supercooling. "...the tendency toward instability should increase as the In content of the melt at the interface increases, as the temperature gradient decreases, and as the freezing rate increases. All three of these trends occurred as solidification proceeded." (1, p. 370) However, a much longer length of stable growth was achieved by horizontal processing. "There were two reasons for this, both related to free-convective strirring. The melt-back was greater with horizontal processing.... This caused the interfacial temperature gradient to be larger since the distance from the interface to the heat sink was decreased. Secondly, the free-convective stirring of the melt decreased the In concentration in the melt at the interface, which was also reflected in the difference in macroscopic concentration

profiles...." (1, p. 370)

Additional information, including plots of concentration versus sample length, on (1) the composition variations within the samples, (2) the influence of Marangoni convection on the solidifying material and (3) the influence of the birefringence effect, can be found in the references listed below.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Alloys, Directional Solidification, Resistance Heating, Single Crystals, Semiconductors, Electronic Materials, Solid Solution, Ternary Systems, Thermal Gradient, Thermal Soak, Supercooling, Solid/Liquid Interface, Sample Homogeneity, Dendritic Structure, Nucleation, Buoyancy-Driven Convection, Sedimentation, Two-Phase System, Mushy Zone, Particle Dispersion, Composition Variation, Surface Morphology, Voids, Grain Boundaries, Twin Boundary, Cracks, Defect Density, Birefringence, Wetting, Non-Wetting of Container, Surface Tension, Sample Detachment from Crucible, Free Surface Solidification, Bubbles, Bubble Formation, Planar Solidification Interface, Interface Stability, Cooling Rate, Volume Change, Marangoni Convection, Contamination Source, Coated Surfaces, Vacuum

Number of Samples: six (three per flight)
Sample Materials: indium-gallium antimonide, $In_xGa_{1-x}Sb$, where x = 0.5, 0.3, 0.1
(In*Ga*Sb*)
Container Materials: carbon coated silica
(Si*O*, C*)

Experiment/Material Applications:
Group IV and III-V semiconductor alloys have a wide variety of useful properties. However, large sized, homogeneous single crystals of the concentrated alloys are not produced because of problems with twin and grain boundaries. It had been theorized that, on Earth, compositional variations caused by convective flows contribute to grain formation in the solidified material. To test this theory, experiments were performed under low-gravity conditions to eliminate or reduce these flows.

Although InSb-GaSb alloys have potential uses, they are also convenient models of hazardous materials such as HgCdTe and PbSnTe.

References/Applicable Publications:

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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt Date: December 1973-January 1974 (months experiment was performed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Mixed III-V Crystal Growth (M563); Directional Solidification of InSb-GaSb Alloy Semiconductors

W.R. Wilcox conducted two crystal growth experiments during the Skylab program, one on SL-3 and one on SL-4. Because applicable references combined the results of these experiments, both the SL-3 and SL-4 summaries were combined under Wilcox, Skylab 3 (this chapter).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Alloys, Directional Solidification, Resistance Heating, Single Crystals, Semiconductors, Electronic Materials, Solid Solution, Ternary Systems, Thermal Gradient, Thermal Soak, Supercooling, Solid/Liquid Interface, Sample Homogeneity, Dendritic Structure, Nucleation, Buoyancy-Driven Convection, Sedimentation, Two-Phase System, Mushy Zone, Particle Dispersion, Composition Variation, Surface Morphology, Voids, Grain Boundaries, Twin Boundary, Cracks, Defect Density, Birefringence, Wetting, Non-Wetting of Container, Surface Tension, Sample Detachment from Crucible, Free Surface Solidification, Bubbles, Bubble Formation, Planar Solidification Interface, Interface Stability, Cooling Rate, Volume Change, Marangoni Convection, Contamination Source, Coated Surfaces, Vacuum

Number of Samples: See Wilcox, Skylab SL-3
Sample Materials: See Wilcox, Skylab SL-3
Container Materials: See Wilcox, Skylab SL-3

Experiment/Material Applications:
See Wilcox, Skylab SL-3

References/Applicable Publications:
In addition to the publications listed under Wilcox, SL-3, these publications are also relevant to experiments performed during the two Skylab missions:

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(6) Input received from Principal Investigator W. R. Wilcox, December 1987, September 1988, August 1989, and August 1993.

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt Date: September 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Halide Eutectic Growth (M564)

When certain binary, eutectic mixtures solidify, one of the two phases can solidify in the form of rods or platelets. The eutectic composition of NaCl-NaF solidifies into crystalline rods of sodium fluoride. These NaF rods are transparent and could be used as fiber optic materials if they could be grown without defects. However, on Earth, these structures form imperfectly because of convective flows within the melt. Reportedly, in space, such convective flows are significantly reduced and a continuous, fault-free solidified structure may result.

This Skylab SL-3 experiment was the first in a series of investigations designed by A. S. Yue et al. to study eutectic growth under low-gravity conditions. The specific objective of the experiment was to produce a highly continuous, controlled fiber-like, NaCl-NaF eutectic structure by directional solidification.

Prior to flight, three NaCl-21 wt.% NaF samples (0.31 inch diameter, 2.5 inches long) were prepared. Each sample was contained in a machined, graphite tube and loaded into a stainless steel container. After loading, the interior of the stainless steel container was coated with a graphite paste to prevent reaction of the molten salt with the container wall. A calcined graphite disk was placed over the ingot and a stainless steel cap (with a vent hole) was welded onto the cartridge. The three steel containers were configured within the Skylab Multipurpose Electric Furnace System (MEFS).

During the low-gravity phase of the mission, all but approximately 1/2 inch of each ingot was purposely melted. (The unmelted section of the ingot acted as a seed crystal.) The sample was then directionally solidified at a rate of 0.6 ^{o}C/min and a thermal gradient of 50 ^{o}C/cm. Samples were similarly processed on Earth for comparison.

Post-flight examination of the samples indicated that no reaction between the salt ingots and the graphite occurred. It was also noted that, at the beginning of the solidification process, the NaF eutectic fibers grew perpendicular to the growth direction. This behavior was attributed to improper insulation design, which permitted heat flow in the radial direction. After about 0.12 cm of growth, the fibers were aligned parallel with the growth direction.

Microscopic examination of the samples indicated that the aligned fibers were regularly spaced as well as parallel with the growth direction. It was also reported that the fibers were more continuous (fewer defects) than those found in the Earth-processed material. This result was attributed to the lack of gravity-induced convective flows and vibration normally present during solidification processes on Earth.

Examination of the infrared transmittance properties also revealed the improved continuity of the space-grown fiber. The transmittance was better for the low-gravity sample than for the 1-g processed material throughout the infrared wavelength range investigated.

Results from this experiment led to the following conclusions:

(1) The ability to produce aligned eutectic NaF fibers in the low-gravity environment was due to the absence of convective flows in the liquid during solidification.

(2) Larger transmittance over a wider wavelength was achieved by the low-gravity material because of better alignment of NaF fibers compared to the 1-g processed material.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Eutectics, Fiber Eutectics, Binary Systems, Molten Salts, Fiber Optic Applications, Optics Applications, Two-Phase System, Seed Crystals, Thermal Gradient, Asymmetric Temperature Field, Solidification Rate, Solid/Liquid Interface, Surface Morphology, Fibers, Inter-Fiber Spacing, Single Grain Eutectics, Defects, Defect Density, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Crucible Effects, Material Interaction With Containment Facility, Coated Surfaces, Infrared Transmission, Optical Transmission

Number of Samples: three
Sample Materials: Eutectic salt composition containing 79% sodium chloride and 21% sodium fluoride.
(Na*Cl*, Na*F*)
Container Materials: Coated graphite sleeve in a stainless steel cartridge.
(C*)

Experiment/Material Applications:
NaF rods are transparent and could be used as fiber optic materials if they could be grown without defects.

References/Applicable Publications:
(1) Yue, A. S. and Yu, J. G.: Halide Eutectic Growth. In Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. I, April 30-May 1, 1974, M-74-5, NASA Marshall Space Flight Center, Alabama, pp. 469-474. (post-flight)

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(7) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

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Experiment Origin: USA
Mission: ASTP
Launch Date/Expt Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Docking Module Payload
Processing Facility: Multipurpose Electric Furnace: a gradient furnace located in the ASTP docking module
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Halide Eutectic Growth (MA-131)

This ASTP experiment was the second in a series of investigations designed by A. S. Yue et al. to study eutectic growth under low gravity conditions (see A. S. Yue, Skylab SL-3). The major objective of the experiment was to prepare a superior, fiber-like NaCl-LiF eutectic. The eutectic was to exhibit continuous LiF fibers within the NaCl matrix. Other objectives of the research included (1) determining the continuity of the low-gravity aligned fibers, (2) comparing the fiber continuity of low-gravity and Earth-processed materials, (3) determining the interfiber spacing of low-gravity and Earth-produced LiF fibers, and (4) measuring the transmittance (as a function of wavelength) of the low-gravity and Earth-processed materials.

During the ASTP mission, the Multipurpose Electric Furnace was used to directionally solidify three NaCl-29 wt% LiF eutectic samples. The experimental setup and procedure were the same as the earlier Skylab SL-3 experiment and are described under A. S. Yue, Skylab SL-3.

Post-flight examination of the ingots indicated that all but 0.28 cm of one sample was not melted and all but 0.51 cm of the other two samples was not melted. This result indicated "...a slight offset in the positioning of the heat zones in the furnace." (1, p. 493) It was noted that at the beginning of the solidification process, the LiF fibers grew perpendicular to the direction of solidification. This result was attributed to the heat extraction direction being perpendicular to the ingot axis. After a short distance, however, the fibers were aligned parallel to the growth direction. (This initial misdirection of the fibers was also observed during the Skylab experiment and was attributed to

an improper insulation design of the Skylab furnace.)

Microstructural examination of the low-gravity samples revealed a continuous and straight fiber structure. In contrast, those ingots processed on Earth contained discontinuous and randomly oriented fibers. These differences were attributed to the lack of gravity-induced convective flows that tend to break up and randomly orient the fibers. Measurements of the inter-fiber distance for all samples indicated little difference. The inter-fiber spacing for the Earth-processed material was slightly smaller, which was attributed to the faster cooling rate of these samples during growth.

Image transmission properties of the space-processed materials (sectioned samples) were investigated. "The transmitted image has the same dimensions as the source, an indication that the LiF fibers are perpendicular to the plane of the paper [containing the source]. However, the transmitted image is not as clear as the original image of the source; therefore, a loss of light through transmission is indicated. Some loss is common to all fiber optic materials...." (1, p. 497) The image transmitted through the Earth-processed sample was unclear and the dimensions of the image were enlarged, which is "...an indication that the fibers are diverged away from the source." (1, p. 497)

Far-field infrared transmission curves of transverse sections of low-gravity and 1-g samples indicated that the space-processed samples have a higher transmittance over nearly the entire wavelength range investigated. This indicated that the ASTP samples contained a higher percentage of fibers aligned to the growth direction.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Eutectics, Fiber Eutectics, Molten Salts, Fiber Optic Applications, Optics Applications, Two-Phase System, Seed Crystals, Thermal Gradient, Cooling Rate, Solid/Liquid Interface, Surface Morphology, Fibers, Inter-Fiber Spacing, Single Grain Eutectics, Defects, Defect Density, Buoyancy-Driven Convection, Buoyancy Effects Diminshed, Crucible Effects, Coated Surfaces, Infrared Transmission, Optical Transmission, Furnace Malfunction

Number of Samples: three
Sample Materials: Eutectic salt composition containing 71% sodium chloride and 29% lithium fluoride.
(Na*Cl*, Li*F*)

Container Materials: Coated graphite sleeve in a stainless steel cartridge.

Experiment/Material Applications:
See A. S. Yue, Skylab SL-3.

References/Applicable Publications:
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Affiliation(s): (1,2) During Skylab: Texas Instruments, Dallas, Texas, Currently: (1) Advanced Micro Devices, Sunnyvale, California; (2) INTEL CORP, Santa Clara, California

Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt Date: September 1973
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA), Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Microsegregation in Germanium (M559)

Micro-inhomogeneities of a solute, which occur during the solidification process, can result in resistivity variations in semiconductor materials. These variations can occur both parallel and perpendicular to the growth direction of the crystal and can adversely affect semiconductor performance. The major contribution to solute micro-segregation is gravity-induced convective flow. However, the magnitude of the effect of convective mixing on segregation is not presently understood.

The objectives of this Skylab experiment were to (1) determine if an improvement in solute microsegregation can be achieved during low-gravity solidification and (2) determine if the improved solute microsegregation can be quantified.

During the Skylab SL-3 mission, the Multipurpose Electric Furnace System (MEFS) was used to directionally solidify three single crystal germanium rod samples:

(1) Ge doped with gallium (Ga, 7.8×10^{16} atoms/cc),

(2) Ge doped with antimony (Sb, N-type electron donor, 0.4×10^{15} atoms/cc), and

(3) Ge doped with boron (B, P-type electron acceptor, 2×10^{15} atoms/cc).

Two additional sets of similar samples were prepared for ground-based testing (horizontal and vertical growth directions). All of the samples were prepared from powdered Ge and dopant and

grown in the <111> direction. Each resulting crystal was then enclosed in a graphite tube and encapsulated in an evacuated stainless steel ampoule.

All samples were processed under similar thermal conditions. Initially, the portions of the samples within the furnace hot zone were heated to 1000 ^{o}C within 3 hours (the melting point of Ge is 938 ^{o}C). The samples were then held at this temperature for approximately 2 hours to achieve steady-state conditions. Cool-down (at a rate of 0.6 ^{o}C/min) was initiated by controlled reduction of the power level.

Post-flight examination of the space-processed samples revealed that the B- and Sb-doped crystals became contaminated. "Unfortunately, the amount of contaminants which entered the Ge melt during the 'oven-on' cycle was large enough to interfere with the electrical properties of both Sb and B doped Ge samples.... The major constituents of the contaminants in samples taken from the melt ends of the crystals were shown by emission spectroscopy to be Mn, Cu, and Fe. As the graphite tubes did not contain such large quantities of these transition metals... it is deduced that they entered the melt from the stainless tube during the oven-on cycle." (1, p. 385)

Analysis of the segregation in the Ga-doped Ge sample was conducted with a spreading resistance probe (see Reference (1) for procedure details). It was reported that the low-gravity processed sample contained much less radial segregation than those similar materials processed on Earth. However, all samples showed a significant increase in dopant level near the surface. The Skylab sample's diameter decreased in the regrowth region. This decrease resulted in a free surface and, therefore, surface tension-driven flow may have caused the dopant increase in this area. The 1-g samples had no free surfaces. However, the radial segregation in the Earth-processed material may have been driven by conventional convection caused by a radial thermal gradient.

Examination of the micro-segregation of dopant (resolution of resistance measurements was 5 microns) indicated that the amplitude of the fluctuations exhibited by the space sample was significantly less than in the 1-g material. "The degree of microsegregation (mean square value of these fluctuations over a 100... [micron] interval) was 0.4 percent for the space-grown crystal compared with 1.6 percent and 2.2 percent, respectively, for the ground control samples." (4, p. 58)

These micro- and macrosegregation measurements led to the conclusion that "...solidification in space can provide six-fold improvement in macrosegregation and nearly two-fold improvement in microsegregation for crystal growth by the gradient freeze

method." (1, p. 385)

The effective segregation coefficient was determined (see Reference (1) for details). Reportedly, for the low-gravity sample, k_{eff}, was higher at the solid-liquid interface. This result suggested that the solute boundary layer at the growth interface is larger under low-gravity conditions than on Earth.

<Note: Reference (2) (below) was not used in the preparation of this experiment summary.>

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Single Crystals, Dopant, Semiconductors, Semiconductor Applications, Electronic Materials, Electrical Properties, Thermal Gradient, Thermal Soak, Cooling Rate, Solid/Liquid Interface, Surface Morphology, Segregation, Macrosegregation, Microsegregation, Segregation Coefficient, Axial Segregation, Buoyancy-Driven Convection, Free Surface, Surface Tension-Driven Convection, Sample Detachment from Crucible, Volume Change, Sample Necking, Contamination Source, Crucible Effects, Vacuum

Number of Samples: three
Sample Materials: (1) germanium doped with gallium; (2) germanium doped with antimony; (3) germanium doped with boron
(Ge*Ga*, Ge*Sb*, Ge*B*)
Container Materials: Graphite tube contained in stainless steel ampoule.

Experiment/Material Applications:
It was reported that Ge was selected for this experiment instead of silicon because of its lower melting point. Ge has also been extensively studied on Earth.

Ga, Sb, and B are dopants of Ge. They were selected for this experiment because they have substantially different segregation coefficients in Ge. Since the experiment was designed to study the effect of low-gravity on segregation, these dopants provide an ideal vehicle for characterization. (11)

References/Applicable Publications:

(1) Yue, J. T. and Voltmer, F. W.: Influence of Gravity-Free Solidification on Microsegregation. In Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. I, April 30-May 1, 1974, M-74-5, NASA Marshall Space Flight Center, Alabama, pp. 375-424. (post-flight)

(2) Yue, J. T. and Voltmer, F. W.: Influence of Gravity-Free Solidification on Solute Microsegregation. Journal of Crystal Growth, 29 (1975), pp. 329-341. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(4) Naumann, R. J. and Herring, H. W.: Experiment M559, Influence of Gravity Free Solidification on Microsegregation. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, p. 58. (post-flight)

(5) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(6) Experiment M559-Microsegregation in Germanium. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, p. 5-65. (post-flight)

(7) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(8) Microsegregation in Germanium (M559). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-50. (post-flight; very short summary)

(9) Wiedemeier, H.: Crystal Growth in Microgravity - An Overview. In Applications of Space Flight in Materials Science and Technology, proceedings of a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977 (issued September 1978), pp. 25-39. (post-flight)

(10) Naumann, R, J. and Mason, D.: Influence of Gravity-Free Solidification on Microsegregation. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 26-27. (post-flight)

(11) Input received from Principal Investigator J. T. Yue, July 1993.

(12) Input received from Principal Investigator F. W. Voltmer, July 1993.

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Experiment Origin: USA
Mission: This experiment was to be performed during Skylab, SL-4, Third Skylab Manned Mission.
Launch Date/Expt. Date: The experiment was not performed as scheduled due to insufficient crew time.
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: On-Board Food Freezer
Builder of Processing Facility: Unknown

Experiment:
Ice Formation (SD17 TV112)

This Skylab science demonstration was designed to study the low-gravity formation of ice. The first objective of the demonstration was to observe the freezing of a water drop on a frozen ice cube. General differences between Earth and space solidification were to be determined from photographs which documented the terrestrial and Skylab solidification. It was believed that in the low-gravity environment the following characteristics would be altered: (1) the expansion nature of the freezing liquid and (2) the solutal and thermal convection.

The second objective of the demonstration was to observe the solidification of two water globules suspended on a string inside the freezer. One of the globules was to consist of pure, clear water; the other globule was to consist of dyed water.

The distribution of micro-gas bubbles in the solid-liquid interface was of interest in both experiments. (Bubble generation generally occurs in ice at the interface as a function of solidification rate, impurity level and dissolved gas concentration.)

It was reported that because of a lack of crew time, the planned demonstration could not be performed.

Key Words: Crystal Growth From the Melt, Solidification, Solidification Rate, Aqueous Solutions, Drops, Freezing, Ice, Containerless Processing, Volume Expansion, Gas Formation, Bubbles, Bubble Formation, Bubble Distribution, Interface Phenomena, Impurities, Liquid/Gas Interface, Solid/Liquid Interface, Thermosolutal Convection, Sample Not Processed as Planned

Number of Samples: not applicable
Sample Materials: water, dye
Container Material: not applicable

Experiment/Material Applications:
The specific applications of this research were not discussed in the available publications.

References/Applicable Publications:
(1) Ice Formation (SD17-TV112). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-88.

(2) TV112 - Ice Formation. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, p. 7-44.

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(4) Input received from Principal Investigator B. Facemire, July 1993.

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Co-Investigator(s): Kattamis, T. Z. (Co-Investigator was not his specific title) (2)
Affiliation(s): (1) Grumman Aerospace Corporation, Bethpage, New York; (2) University of Connecticut, Storrs, Connecticut

Experiment Origin: USA
Mission: SPAR 1
Launch Date/Expt. Date: December 1975
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Furnace facility for directional solidification of transparent samples
Builder of Processing Facility: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Contained Polycrystalline Solidification (74-37)

During the solidification of a casting, two types of grain structure may form: (1) equiaxed (roughly spherical grains of nearly the same size) and (2) columnar (cylindrical grains). A zone of equiaxed grain structure will form if (1) nuclei for equiaxed grains are present throughout the melt and (2) growth of the equiaxed grains is greater than that of the columnar grains. Both of these conditions (presence of nuclei and growth of eqiaxed grains) are influenced by gravity. For example, gravity-induced convection (driven by composition gradients that arise from rejection of solute at the solidification interface) can contribute to the formation of an equiaxed grain structure.

Metals melting experiments performed in the low-gravity environment such as (1) Skylab experiment M551 (see Poorman, Skylab SL-2 (Chapter 14)) and (2) drop tower experiments illustrated an unexpected occurrence of fine-grained equiaxed microstructures in the cast samples. These structures were formed despite the reduction of gravity-induced convection. Therefore, further investigation of the gravitational effects on columnar-to-equiaxed transition was initiated.

This SPAR 1 experiment was the first in a series of investigations designed by Papazian et al. to study the low-gravity solidification behavior of a polycrystalline material. (The experiment also represented one of two investigations performed by Papazian during the SPAR 1 sounding rocket mission (see Papazian, SPAR 1, Experiment 74-36 (Chapter 12)).) The objectives of Experiment 74-37 were to (1) directly measure the effect of reduced gravity on the width of the solute enriched zone ahead of a

solidification interface and (2) investigate the columnar-to-equiaxed transition during polycrystalline solidification.

In preparation for the experiment, a sample of transparent cyclohexanol mixed with fluorescein dye was placed in a silica cuvette. (Use of this material allowed the observation of the buildup of the colored solute (fluorescein, green in color) ahead of the solidification interface.) During the mission, solidification of the material was accomplished via a thermoelectric cooling device which was placed at the bottom of the cuvette. Reflected illumination from a tungsten filament microscope illuminator provided lighting for 35 mm photographic recording of the process.

It was intended that a sequence of 220 photographs would record the solidification process and buildup of solute. "Unfortunately, during flight, the government furnished (GFE) camera for this experiment malfunctioned. We obtained 4 test exposures before lift-off, followed by 13 exposures which seem to have been taken at the beginning of the low-gravity interval and 56 exposures which were probably taken toward the end of the cooling sequence. The first 14 low-gravity frames show that no crystallization had yet occurred; this is consistent with our ground-based results in which the first solid is observed at approximately 20 s. The 56 subsequent frames show approximately 6 mm of solid present...." (1, p. VIII-9)

Further post-flight examination of the photographs revealed that the solute-enriched zone ahead of the interface was wider and more irregular than that observed for ground-based tests. The solidified flight sample consisted of an equiaxed grain structure, while the 1-g processed samples consisted of a columnar grain morphology. The grain size of the flight sample was 1.2 mm. The grain sizes of the ground-processed samples (width of columnar grain) ranged between 2 and 3.5 mm. The average growth rate of the flight sample was reported as 60 microns/sec which was significantly higher than the growth rate of the ground samples (20 to 30 microns/sec).

The smaller grain size, equiaxed grain morphology, and a larger average macroscopic growth rate of the flight sample were attributed to parasitic nucleation ahead of the interface (that was not observed in the ground-based samples). Several possibilities for this different behavior were reported: (1) the different thermal histories of the flight and ground-processed samples, (2) contamination of the flight sample, (3) perturbation of the heat and fluid flow caused by the presence of a small bubble in the flight sample, (4) launch-induced fluid motion, and (5) inadequate superheating of the flight sample prior to launch. Subsequent ground-based investigations led to the conclusion that

the parasitic nucleation "...may be attributed to ordered islands within the liquid, which survived remelting because of the low degree of superheating (approximately 1.5 ^{o}C), and did not settle because of reduced gravity and acted as nuclei during cooling." (1, p. VIII-19)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Model Materials, Transparent Liquids, Grain Structure, Grain Size, Columnar to Equiaxed Transition, Solutal Gradients, Nucleation, Surface Morphology, Solid/Liquid Interface, Rejection of Solute at Interface, Solidification Front Physics, Interface Physics, Interface Phenomena, Growth Rate, Heat and Mass Transfer, Bubbles, Bubble Formation, Superheating, Casting, Buoynacy-Driven Convection, Contamination Source, Rocket Motion, Launch-Induced Fluid Motion, Acceleration Effects, Photographic Difficulties

Number of Samples: one
Sample Materials: cyclohexanol-fluorescein ($C_6H_{11}OH$ - $C_{20}H_{12}O_5$)
(C*H*O*)
Container Materials: silica
(Si*O*)

Experiment/Material Applications:
"The unique and severe restrictions of the sounding rocket as a research vehicle and the desire to glean as much information as possible from a 300 s experiment led... to the choice of low melting point, low entropy of fusion organic materials as models for metallic solidification. These materials have been used successfully in the past and have the advantage of low melting point [22 ^{o}C], ease of handling, and transparency." (1, p. VIII-3)

References/Applicable Publications:
(1) Papazian, J. M. and Kattamis, T. Z: Contained Polycrystalline Solidification in Low Gravity. In Space Processing Applications Rocket Project, Spar I Final Report, NASA TM X-3458, pp. VIII-1 -VIII-20, December 1976. (post-flight Report)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Input received from Principal Investigator J. M. Papazian, December 1987, September 1988, and August 1993.

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Experiment Origin: USA
Mission: SPAR 4
Launch Date/Expt. Date: June 1977
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Furnace facility with radial cooling
Builder of Processing Facility: Grumman Aerospace Corporation, Bethpage, New York

Experiment:
Contained Polycrystalline Solidification in Low G (74-37)

This SPAR 4 experiment was the second in a series of investigations designed by Papazian et al. to study the low-gravity solidification behavior of a polycrystalline material (see Papazian, SPAR 1, Experiment 74-37). The specific objective of the experiment was to study the columnar-to-equiaxed transition (CET) during polycrystalline solidification.

Prior to the mission, two samples of 30 wt.% NH_4Cl in water (liquidus temperature of 33 ^{o}C) and two samples of 37 wt.% NH_4Cl in water (liquidus temperature of 67 ^{o}C) were prepared. The experimental apparatus consisted of (1) a sample chamber configured with four independent, semi-cylindrical pockets contained between Plexiglas^{TM} faces, (2) an electronics box, (3) a motor-driven camera (250 exposures), (4) a freon reservoir designed to deliver freon for sample quench, (5) a support structure, and (6) heaters.

Forty-five minutes prior to launch, the heaters were turned on and the sample chamber temperature was maintained at 90 ^{o}C. At lift-off, power to the heaters was severed. About 30 seconds after the start of the low-gravity period, the freon quench was initiated and filming of the experiment took place.

It was reported that "...a partial blockage in the needle valve of the quenching system prevented solidification of the samples. Thus no data on the effect of gravity on the CET were obtained. Incomplete preflight melting of the NH_4Cl was also observed. It was established that slow solidification, a long waiting time at room temperature, and the presence of agitation during this time coarsened the room temperature structure of the solid and lengthened the time required for remelting." (1, p. III-ii)

The results of preflight, ground-based experiments are reported in Reference (2).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Model Materials, Aqueous Solutions, Grain Structure, Columnar-to-Equiaxed Transition, Surface Morphology, Solid/Liquid Interface, Solidification Front Physics, Interface Physics, Interface Phenomena, Quench Process, Hardware Malfunction, Processing Difficulty

Number of Samples: four
Sample Materials: Samples 1 and 3: 30 wt.% NH_4Cl in water; Samples 2 and 4: 37 wt.% NH_4Cl in water
(N*H*Cl*H*O*)
Container Materials: Plexiglas™

Experiment/Material Applications:
"Solutions of NH_4Cl in H_2O were selected as sample materials, because this system has been used successfully by others in previous experiments." (1, p. III-2)

References/Applicable Publications:
(1) Papazian, J. M., Kesselman, M., and Kattamis, T. Z.: Flight IV Technical Report for Experiment 74-37 Contained Polycrystalline Solidification in Low-G. In Space Processing Applications Rocket Project SPAR IV-Engineering Report (Final), NASA TM-78235, pp. III-I - III-20, January 1980. (post-flight Report)

(2) Papazian, J. M. and Kattamis, T. Z.: Flight IV Technical Report for Experiment 74-37 Contained Polycrystalline Solidification in Low G. Grumman Corporate Research Center Report RM-645, December 1977, 20 pp. (ground-based research included)

(3) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(4) Contained Polycrystalline Solidification in Low Gravity. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, edited by R. J. Naumann, January 1979, NASA TM-78217, pp. 19-20. (post-flight)

(5) Input received from Principal Investigator J. M. Papazian, December 1987, September 1988, and August 1993.

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Experiment Origin: USA
Mission: SPAR 5
Launch Date/Expt. Date: September 1978
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Furnace facility with radial cooling
Builder of Processing Facility: Grumman Aerospace Corporation, Bethpage, New York

Experiment:
Contained Polycrystalline Solidification in Low Gravity (74-37)

This SPAR 5 experiment was the third in a series of experiments designed by Papazian et al. to study the low-gravity solidification behavior of a polycrystalline material (see Papazian, SPAR 1, Experiment 74-37, and SPAR 4). The specific objective of this experiment was to study columnar-to-equiaxed transition (CET) during polycrystalline solidification.

Prior to the mission, two solutions of NH_4Cl-H_2O were prepared: (1) 325 grams/liter NH_4Cl (liquidus temperature of 35 ^{o}C) and (2) 362.5 grams/liter H_4Cl (liquidus temperature of 55 ^{o}C). (Two samples of each solution were prepared for the experiment.)

The experimental apparatus consisted of (1) a sample chamber of four independent, semi-cylindrical pockets contained between Plexiglas™ faces, (2) an electronics box, (3) a motor-driven camera (230 exposures, 1 frame/second), (4) a freon reservoir designed to deliver freon for sample quench, (5) a support structure, and (6) heaters (details of the experimental apparatus may be located in Reference (1)).

Prior to launch, the sample chamber was heated and maintained at a temperature of 80 ^{o}C. At launch, the heaters were switched off. One hundred seconds after launch (low-gravity conditions), a freon sample quench was initiated and solidification occurred throughout the next 200 seconds. During this time the solidification process was photographed. Corresponding ground-based studies were performed for comparison.

Post-flight examination of the flight samples' thermal data indicated "...that because of reduced convective transport of heat, the liquid portion of the sample was significantly warmer. This

led to a steeper thermal gradient in the liquid, but more significantly, this did not allow for the growth of any equiaxed grains ahead of the columnar interface. No gravity driven convective mechanisms occurred to transport nuclei into the liquid, but this is thought to be of secondary significance. Even if nuclei had been present the thermal conditions would not have allowed them to grow. In order for an equiaxed zone to form, growth of the equiaxed grains must occur faster than growth of the columnar grains. This was not possible in the reduced gravity environment." (1, p. V-17)

Examination of the photographic records and solidified samples led to the following conclusions:
(1) Significant convection driven by thermal inversion was absent in the flight experiment.
(2) Grain multiplication by showering (growth of dendritic fragments at the open surface of an ingot which shower through the melt and settle on the columnar interface) was not observed.
(3) Grain multiplication, driven by either thermal inversion or compositional inversion, did not occur.
(4) Completely columnar structures were obtained in all flight samples, while the ground-processed samples exhibited equiaxed structures making up 25% to 100% of the cast material.
(5) In the flight samples, (a) the thermal gradient was steeper and (b) the rate of cooling of the liquid was slower than those observed in the 1-g samples.
(6) The crystalline growth rate was not changed by low-gravity processing.
(7) Bubbles were not pushed by the advancing solidification front.
(8) An equiaxed structure did not result from an induced "big bang" nucleation process (see Reference (1)).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Thermal Gradient, Model Materials, Aqueous Solutions, Grain Structure, Grain Size, Columnar to Equiaxed Transition, Solutal Gradient, Nucleation, Surface Morphology, Solid/Liquid Interface, Solidification Front Physics, Interface Physics, Interface Phenomena, Dendritic Structure, Cooling Rate, Bubbles, Casting, Buoynacy-Driven Convection, Buoyancy Effects Diminished, Quench Process, Thermal Environment More Extreme Than Predicted

Number of Samples: four

Sample Materials: $NH_4Cl:H_2O$ solutions, two compositions used, (1) 325 gm/l NH_4Cl in water, (2) 362.5 gm/l NH_4Cl in water (N*H*Cl*H*O*)
Container Materials: Plexiglas™

Experiment/Material Applications:
Any polycrystalline solidification process.
See Papazian, SPAR 4.

References/Applicable Publications:
(1) Papazian, J. M. and Kattamis, T. Z.: Contained Polycrystalline Solidification in Low-G. In Space Processing Applications Rocket Project, SPAR V Final Report, NASA TM-78275, pp. V-1 - V-36, August 1980. (post-flight analysis)

(2) Papazian, J. M. and Kattamis, T. Z.: Effect of Reduced Gravity on the Solidification Microstructures of NH4CL: H2O Alloys. Metallurgical Transactions A, Vol. 11A, March 1980 pp. 483-93.

(3) Papazian, J. M. and Kattamis, T. Z: SPAR V Technical Report for Experiment 74-37 Contained Polycrystalline Solidification in Low-G. Grumman Corporate Research Center Report RE-569, February 1979, 29 pp. and addendum, May 1979, 4 pp.

(4) Naumann, R. J. (editor): Contained Polycrystalline Solidification in Low Gravity. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, NASA TM-78217, January 1979, pp. 19-20. (post-flight)

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Experiment Origin: USA
Mission: SPAR 3
Launch Date/Expt. Date: December 1976
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Furnace Facility
Builder of Processing Facility: Rockwell International Science Center, Thousand Oaks, California

Experiment:
Epitaxial Growth of Single Crystal Film (74-45)

The Liquid Phase Epitaxy (LPE) crystal growth technique involves ". . . bringing a substrate crystal into brief contact with a melt or molten solution near its melting point." (1, p. V-3) Reportedly, the "Low-g environment is of particular benefit to LPE because of the elimination of convection during crystal growth, as well as simplification of the problem of initial homogenization of the molten solution prior to initiating crystal growth. Modest improvements in crystal quality can can have significant technological and economic importance." (1, p. V-3)

This SPAR 3 experiment was the first in a series of investigations designed by Lind et al. to study crystal growth by LPE under low-gravity conditions. The specific objective of the experiment was to grow an epitaxial film of gallium arsenide (GaAs). <Note: Somewhat related solution growth experiments were performed by Lind and/or Nielsen et al. during the ASTP, Spacelab 1, and LDEF missions (Chapter 8).>

Prior to the SPAR 3 mission, two GaAs wafers were mounted in a graphite slider. During the rocket flight, the slider (which was driven by a piston) was to move the wafers until they contacted a high temperature saturated solution (approximately 720 ^{o}C) of GaAs in liquid Ga. At the end of 1 minute of growth the slider was to be retracted.

It was reported that "...the furnace and its control system worked properly until the slider mechanism with the substrate was moved into contact with the growth solution. The temperature readings became erratic at that time making it difficult to determine what [the] temperature was near the growth solutions.

Post-flight inspection showed that the slider mechanism was broken during substrate contact with the solution. When the slider mechanism was withdrawn, it failed to remove the substrate from the growth solution." (1, p. V-3).

No results related to the sample were obtained. However, in relation to the processing facility, it was determined that:

(1) It was necessary to reinforce graphite parts with stainless steel.

(2) Thermocouples had to be electrically isolated from graphite parts.

Key Words: Crystal Growth From the Melt, Liquid Phase Epitaxy, Single Crystals, Binary Systems, Saturated Solution, Substrates, Semiconductor Applications, Semiconductors, Electronic Materials, Optics Applications, Sample Homogeneity, Films, Thin Films, Solid/Liquid Interface, Buoyancy-Driven Convection, Piston System, Hardware Malfunction

Number of Samples: two
Sample Materials: gallium-arsenide (Ga*As*)
Container Materials: Sample holder: graphite (C*)

Experiment/Material Applications:
Please refer to Lind, SPAR 6.

References/Applicable Publications:
(1) Lind, M. D.: Epitaxial Growth of Single Crystal Film. In Space Processing Applications Rocket Project, SPAR III-Final Report, NASA TM-78137, pp. V-1 - V-4, January 1978. (post-flight)

(2) Toth, S. and Frayman, M: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

(3) Naumann, R. J. (editor): Epitaxial Growth of Single Crystal Films. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, NASA TM-78217, January 1979, pp. 29-30. (post-flight)

(4) Input received from Principal Investigator M. D. Lind, August 1993.

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Co-Investigator(s): Kroes, R. L. (2), Immorlica, A. A., Jr. (3)
Affiliation(s): (1,3) During SPAR 6: Rockwell International Science Center, Thousand Oaks, California; (1) Currently: Retired; (2) National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (3) Currently: Unknown

Experiment Origin: USA
Mission: SPAR 6
Launch Date/Expt. Date: October 1979
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Liquid Phase Epitaxy (LPE) processor containing a resistance heated furnace, sample slider and retractor
Builder of Processing Facility: Rockwell International Science Center, Thousand Oaks, California

Experiment:
Epitaxial Growth of Single Crystal Films (74-45)

This SPAR 6 experiment was the second in a series of investigations designed by Lind et al. to study crystal growth by Liquid Phase Epitaxy (LPE) under low-gravity conditions (see Lind, SPAR 4). The specific objectives of this experiment were to (1) improve understanding of the LPE crystal growth process and (2) confirm theoretical analysis of material transport and growth kinetics during the process.

The SPAR 6 experimental apparatus consisted of (1) a tubular resistance heated (nichrome wire) furnace, (2) an atmospheric control system (which provided a hydrogen flow inside the furnace to prevent incorporation of oxygen in the crystal), (3) a pheumatically operated, graphite slider (which positioned the two (1.1 cm by 1 m^3 cm) substrates in contact with the solution), and (4) an electronic controller (which provided temperature control and operation of the hydrogen gas system). (Reference (1) contains additional details of the experimental apparatus and preflight testing procedures.)

Prior to launch, two cleaned GaAs wafers were loaded into the slider. The wafers had been cut perpendicular to the 100 direction. Ninety minutes before launch, the supersaturated solution of arsenic in gallium was heated to 1050 K. At launch, the solution temperature was lowered so that the growth temperature of 990 K was reached 4 minutes into the flight. This assured that (1) the correct solution temperature was attained well into the low-gravity period of the mission and (2) the fluid motions induced by launch were fully damped. The slider mechanism was then

activated, bringing the wafers into contact with the solution, allowing a one minute growth period.

Samples were also processed on the ground, using the flight hardware, under identical conditions (with the exception of gravity level).

Post-flight examination of the thermal data indicated that "The recorded temperatures are not precise enough to confirm that the desired supercooling interval, [delta]T = 10^{o}K, was achieved. The reliability of temperature control proven during pre-flight tests is the only evidence available to show that it was." (1, p. IV-13) Examination of the samples revealed that the thickness of those films processed in low gravity was less than those of the ground samples. This result was as expected if only diffusive mass transfer (and not convective transfer) took place during the flight experiment. The actual thickness, 1.5 microns, was in good agreement with theoretical predictions. X-ray diffraction topography indicated the films were single crystalline, of fairly good quality, and of uniform composition.

Reportedly, "...the quality of the flight samples was better than the worst but not as good as the best of the earth processed samples. Thus, with regard to the effects of space processing on the film quality, no definitive conclusions can be drawn from this experiment and further flights would be necessary to answer this question." (1, p. IV-18)

Key Words: Crystal Growth From the Melt, Liquid Phase Epitaxy, Single Crystals, Binary Systems, Saturated Solution, Substrates, Semiconductor Applications, Semiconductors, Electronic Materials, Optics Applications, Sample Homogeneity, Resistance Heating, Supersaturation, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Mass Transfer, Growth Kinetics, Supercooling, Films, Thin Films, Solid/Liquid Interface, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Rocket Motion, Fluid Motion Damping, Thermal Control, Piston System, Launch-Induced Fluid Motion

Number of Samples: two
Sample Materials: gallium-arsenide; processing atmosphere: hydrogen
(Ga*As*, H*)

Container Materials: Sample holder: graphite (C*)

Experiment/Material Applications:
"Gallium arsenide LPE appeared to be an appropriate process for investigation on a SPAR flight for the following reasons: (1) the process variables, including growth temperature and rates, are compatible with the capabilities of the Black Brant VC [sounding rocket] flights; and (2) the numerous important technological applications of gallium arsenide LPE films [e.g. semiconductor devices, optical devices] have stimulated broad interest in this process and material, and therefore there was available ample background information on which to base the design of a space flight experiment, and with which to compare results." (1, p. IV-2)

References/Applicable Publications:
(1) Lind, M. D., Kroes, R. L., and Immorlica, A. A.: Final Post Flight Report on SPAR VI, Experiment No. 74-45 Epitaxial Growth of Single Crystal Films. In Space Processing Applications Rocket Project SPAR VI Final Report, October 1981, NASA TM-82433, pp. IV-i - IV-38. (post-flight)

(2) Input received from Principal Investigator M. D. Lind, August 1993.

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Experiment Origin: Japan
Mission: TT-500A 8 (Materials Processing Flight #1)
Launch Date/Expt. Date: September 1980
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: One electric furnace
Builder of Processing Facility: Unknown

Experiment:
Si-As-Te Amorphous Semiconductor

<Note: Y. Hamakawa conducted five solidification experiments during the TT-500A sounding rocket program (see Hamakawa, TT-500A 8, TT-500A 9, TT-500A 10, TT-500A 11, and TT-500A 12 (all in this chapter)). Reference (1) (as listed below) combined the results from the TT-500A 8 and TT-500A 12 flights. It was, therefore, difficult to determine the differences (if any) between the TT-500A 8 and TT-500A 12 experimental objectives, setup, or results. Information for both these experiments was obtained from Reference (1).>

This TT-500A 8 experiment was the first in a series of investigations designed by Hamakawa to study the low-gravity solidification of a Si-As-Te semiconductor. The specific objectives of the research were to (1) produce a homogeneous, multicomponent, amorphous semiconductor and (2) evaluate and compare low-gravity and 1-g processed semiconductors.

On Earth, processing of the Si-As-Te amorphous semiconductor is difficult because of significant physical property differences between the elemental components. These differences include melting points (Si = 1412 ^{o}C, As = 817 ^{o}C, Te = 449 ^{o}C) and densities (Si = 2.33 g/cm^{3}, As = 5.72 g/cm^{3}, Te = 6.24 g/cm^{3}). Another difficulty in the preparation of the Si-As-Te material is the very high vapor pressure of As (>100 kg/cm^{2} at the eutectic temperature of 1100 ^{o}C). <Note: The reason why the high vapor pressure of this material was a problem was not presented.> The vapor pressure difficulty was addressed during the preflight sample preparation procedure (see Reference (1) for details).

During the TT-500A 8 and TT-500A 12 mssions, an electric furnace was used to melt and resolidify powder samples (100 mesh) of Si-As-Te semiconductors. The samples had been sealed in fused

silica ampoules (75 mm inner diameter, 38 mm long) at a pressure below 10^{-5} Torr.

During the TT-500A 8 mission, the sample material was heated to 1260 ^{o}C and held at that temperature for 161 seconds. The sample was then quenched to below 750 ^{o}C in 120 seconds. <Note: Details of the experimental parameters from the TT-500A 12 mission could not be located.>

Reference (1) reported (neither mission nor samples were specified) that "In the experimental furnace... [capsule], $7.5x10^{-4}$ G for the principal axis and less than $6.0\text{-}7.5x10^{-2}$ G for pitch (Y) and Yo axis were obtained for 360 sec." (1, p. 370) <Note: The definition of "Yo axis" is unclear to the editors.> However, Reference (3) reported (for the TT-500A 8 mission) that "...the yo-yo despinners were released before the payload separation. Therefore the spin rate of the payload section was not sufficiently reduced. It was decreased to only 150 deg/sec by the gas-jets control system. The electric furnaces in the payload section were exposed in the 0.1g environment." (3, p. 2)

Post-flight examination of the ampoules (neither mission nor samples were specified) revealed that "...about one third of the alloyed specimen... [was] evaporated on the wall of the ampoule due to a considerably good wettability to the quartz wall. However, about 2 g, of the solid specimen was fortunately obtained. The reason is presumably due to an existence of gradual gravity during the re-entry process." (1, p. 371)

Comparison of the space- and 1-g processed samples (mission not specified) revealed that the flight sample contained no microvoids and no micro-grains. "Moreover, its cleaved surface has no ordering cleavage faces. This fact shows more direct evidence of the vitreous system." (1, p. 372)

X-ray diffraction, electrical and optical property studies, and differential thermal calorimetry were also performed on the samples. These analyses indicated a more complete amorphous structure for the flight samples (see Reference (1) for details).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Ternary Systems, Alloys, Powders, Amorphous Materials, Electronic Materials, Semiconductors, Semiconductor Applications, Optics Applications, Glass Formation, Density Difference, Wetting, Wetting of Container, Evaporation, Sample Evaporation, Gas Pressure, Surface Morphology, Sample Homogeneity, Solid/Liquid Interface, Eutectics, Voids, Grain Structure, Thermal Soak, Quench Process,

Rotation of Payload, Acceleration Effects, Rocket Despin Failure, Vehicle Re-entry Forces/Vibration

Number of Samples: one
Sample Material: Si-As-Te amorphous semiconductor (composition unknown)
(Si*As*Te*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
"Ternary chalcogenide Si-As-Te system is an interesting semiconductor in views of both basic physics and technological applications. Since Si-As-Te system consists of IV-III-II hedral bonding network, it has a very large glass forming region with a wide physical constant controllability.... For example, [the] energy gap can be controlled in a range from 0.6 eV to 2.5 eV which is corresponding to classical semiconductors Ge (0.66 eV) Si (1.10 eV), GaAs (1.43 eV) and GaP (2.25 eV).... This fact indicates that it would be a suitable system to investigate the compositional dependence of the atomic and electronic properties in the random network of solids." (1, p. 362) Also, from a technological view, Si-As-Te "...could be applied to multi-layered heterojunction devices and also opto-electronic functional elements in a wide spectral region from near infrared to visible light region. Moreover, this material can be deposited on any inexpensive substrates such as glass, ceramics and metal surfaces. This advantage derives a big significance of a good-massproduceability [sic] with low cost device fabrication processes in the future." (1, p. 362)

References/Applicable Publications:
(1) Hamakawa, Y., Okamoto, H., and Sada, C.: Fabrication of Si-As-Te Amorphous Semiconductor in the Microgravity Environment in Space. In 2nd Joint Japan-Germany-ESA Symposium on Microgravity Research, Tokyo, March 25-26, 1985, Summary Report, pp. 361-374. (post-flight)

(2) Sawaoka, A. B. and Kanbayashi, A.: Recent Developments and Future Outlook in Japan. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 13-24. (post-flight information)

(3) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. 34th International Astronautical Federation/International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary)

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Experiment Origin: Japan
Mission: TT-500A 9 (Materials Processing Flight #2)
Launch Date/Expt. Date: January 1981
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: One electric furnace
Builder of Processing Facility: Unknown

Experiment:
Si-As-Te Amorphous Semiconductor

This TT-500A experiment was the second in a series of investigations designed by Hamakawa to study the low-gravity solidification of a Si-As-Te semiconductor (see Hamakawa, TT-500A 8).

A discussion of the specific objectives and equipment configuration of the experiment could not be located.

Although the rocket was successfully launched, it was reported that "...the TT-500A rocket... [was]... not recovered because of its floating system malfunction." (1, p. 2) No further details concerning the system anomaly appear to be available and no evaluation of the experimental data could be located.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Ternary Systems, Alloys, Amorphous Materials, Electronic Materials, Semiconductors, Semiconductor Applications, Optics Applications, Glass Formation, Surface Morphology, Sample Homogeneity, Solid/Liquid Interface, Payload Recovery System Failure

Number of Samples: unknown
Sample Materials: Si-As-Te amorphous semiconductor (composition unknown)
(Si*As*Te*)
Container Materials: unknown

Experiment/Material Applications:
See Hamakawa, TT-500A 8.

References/Applicable Publications:
(1) Kajiwara, K., Matsuda, T., Shibato, T., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. 34th International Astronautical Federation/International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary)

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Experiment Origin: Japan
Mission: TT-500A 10 (Materials Processing Flight #3)
Launch Date/Expt. Date: August 1981
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Three electric furnaces
Builder of Processing Facility: Unknown

Experiment:
Si-As-Te Amorphous Semiconductor

This TT-500A experiment was the third in a series of investigations designed by Hamakawa to study the low-gravity solidification of a Si-As-Te semiconductor (see Hamakawa, TT-500A 8, TT-500A 9).

A discussion of the specific objectives and equipment configuration of the experiment could not be located.

Although the rocket was successfully launched, "...the TT-500A rocket [was] not recovered because... a main parachute... [was]... not activated...." (1, p. 2) No further details concerning this parachute anomaly appear to be available.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Ternary Systems, Alloys, Amorphous Materials, Electronic Materials, Semiconductors, Semiconductor Applications, Optics Applications, Glass Formation, Surface Morphology, Sample Homogeneity, Solid/Liquid Interface, Payload Recovery System Failure

Number of Samples: unknown
Sample Materials: Si-As-Te amorphous semiconductor (composition unknown)
(Si*As*Te*)
Container Materials: unknown

Experiment/Material Applications:
See Hamakawa, TT-500A 8.

References/Applicable Publications:
(1) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. 34th International Astronautical Federation/International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short summary)

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Experiment Origin: Japan
Mission: TT-500A 11 (Materials Processing Flight #4)
Launch Date/Expt. Date: August 1982
Launched From: Takesaki Launch site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: One electric furnace
Builder of Processing Facility: Unknown

Experiment:
Si-As-Te Amorphous Semiconductor

This TT-500A 11 experiment was the fourth in a series of investigations designed by Hamakawa to study the low-gravity solidification of a Si-As-Te semiconductor (see Hamakawa, TT-500A 8, TT-500A 9, and TT-500A 10).

A discussion of the specific objectives and equipment configuration of the experiment could not be located in the published literature.

It appears that during the mission, the electric furnace "... overheated and [the] experiment material evaporated." (1, p. 2) No further details concerning the furnace anomaly appear to be available.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Ternary Systems, Alloys, Amorphous Materials, Electronic Materials, Semiconductors, Semiconductor Applications, Optics Applications, Glass Formation, Surface Morphology, Evaporation, Sample Evaporation, Sample Homogeneity, Solid/Liquid Interface, Thermal Control, Furnace Malfunction

Number of Samples: unknown
Sample Materials: Si-As-Te amorphous semiconductor (composition unknown)
(Si*As*Te*)
Container Materials: unknown

Experiment/Material Applications:
See Hamakawa, TT-500A 8.

References/Applicable Publications:
(1) Kajiwara, K., Matsuda, T., Shibato, T., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket. 34th International Astronautical Federation/International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short description)

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Experiment Origin: Japan
Mission: TT-500A 12 (Materials Processing Flight #5)
Launch Date/Expt. Date: January 1983
Launched From: Takesaki Launch Site in Tanegashima Island (Tanegashima Space Center)
Payload Type: Sounding Rocket Experiment
Processing Facility: Two electric furnaces
Builder of Processing Facility: Unknown

Experiment:
Si-As-Te Amorphous Semiconductor

This TT-500A experiment was the fifth in a series of investigations designed by Hamakawa to study the low-gravity solidification of a Si-As-Te semiconductor (see Hamakawa, TT-500A 8, TT-500A 9, TT-500A 10, and TT-500A 11).

The results from this investigation were combined with those of the TT-500A 8 experiment and published in Reference (1). A summary of the TT-500A 8 and TT-500A 12 experiments on Si-As-Te amorphous semiconductors can be found in Hamakawa, TT-500A 8 (this chapter).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Ternary Systems, Alloys, Powders, Amorphous Materials, Electronic Materials, Semiconductors, Semiconductor Applications, Optics Applications, Glass Formation, Density Difference, Wetting, Wetting of Container, Evaporation, Sample Evaporation, Gas Pressure, Surface Morphology, Sample Homogeneity, Solid/Liquid Interface, Eutectics, Voids, Grain Structure, Thermal Soak, Quench Process

Number of Samples: unknown, probably two
Sample Materials: Si-As-Te amorphous semiconductor (composition unknown)
(Si*As*Te*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
See Hamakawa, TT-500A 8.

References/Applicable Publications:
(1) Hamakawa, Y., Okamoto, H., and Sada, C.: Fabrication of Si-As-Te Amorphous Semiconductor in the Microgravity Environment in Space. In the 2nd Joint Japan-Germany-ESA Symposium on Microgravity Research, Tokyo, March 25-26, 1985, Summary Report, pp. 362-374. (post-flight)

(2) Kajiwara, K., Matsuda, T., Shibato, Y., Masuda, T., and Akimoto, T.: Results of Japanese Space Processing Experiments Using the TT-500A Rocket, 34th International Astronautical Federation/International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-157, 9 pp. (very short description)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 4
Launch Date/Expt Date: May 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 03
Builder of Processing Facility: Deutsche Forschungs-und Versuchsanstalt für Luft-und Raumfahrt (DFVLR), Federal Republic of Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft-und Raumfahrt (DLR).>

Experiment:
Directional Solidification of Doped Semiconductors (four experiments designated as Germanium #1, Germanium #2, Germanium #3, and Germanium #4)

<Note: Walter performed four experiments during this TEXUS 4 mission. However, none of the available publications detailed the major differences between the four experiments. Therefore, the following summary describes the objectives, experimental setup, and results from all four investigations.>

In a low-gravity environment, solidification experiments can be performed under well-defined, steady-state conditions. Such conditions enhance the production of homogeneously doped, single-crystal semiconductors. In contrast, on Earth, compositional fluctuations in the melt cause dopant fluctuations in the sample. These fluctuations produce striations in the solidified material.

This set of four TEXUS 4 experiments was the first in a series of investigations designed by Walter to study the low gravity directional solidification of germanium doped with gallium. The specific objective of the experiments were to determine if directional solidification studies were appropriate for a short-duration sounding rocket flight.

Prior to the rocket launch, four crystals of Ge doped with Ga were prepared. (The concentration of these samples ranged from 10^{17} to 10^{19} Ga atoms/cm^3.) Each of these samples was produced by Czochralski crystal growth ([111] oriented seed). Each sample was enclosed in a crucible such that sample free surfaces were

eliminated (to avoid Marangoni convection induced by surface-tension gradients). Each of these crucibles was placed in its own gradient furnace within the TEXUS Experiment Module TEM 03.

Just prior to lift-off, the samples were partially melted. Solidification was initiated before launch such that approximately 2 cm of 1-g processed material was attained. Solidification continued through the liftoff, the low-gravity period, re-entry, and after rocket impact. Accelerations of between 20 g and 10^{-5} g were experienced by the samples during this time. Approximately 2 cm of material was solidified during the low-gravity period.

It was reported that during the mission, the "...projected values of solidification rate (50 μ/s) and temperature gradient (60 K/cm) were maintained within the order of magnitude to the end of solidification. The cooling rate increased from the initial value of approximately 0.1 K/s to about 0.3 K/s." (4, p. 166)

Post-flight examination of the samples indicated that this processing method resulted in a material which contained dopant striations (as determined by differential etching and variation of electrical properties) which were reflective of the various flight events (g levels). However, no striations were observed in that portion of the material solidified in the low-gravity environment. Steady-state crystal growth was achieved within seconds after low-gravity conditions had been achieved. Reportedly, it appeared that crystal growth was less sensitive to axial accelerations than to radial accelerations.

The results indicated that it was possible to perform directional solidification in a sounding rocket flight and obtain suitable samples for investigation. It was reported, however, that materials selected for such experiments must (1) be able to be processed at high growth rates, and (2) at these high growth rates, still yield quality crystals.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Steady-State Solidification, Thermal Gradient, Seed Crystals, Semiconductors, Electronic Materials, Binary Systems, Solid/Liquid Interface, Free Surface Ellimination, Marangoni Convection, Dopant, Striations, Composition Variation, Sample Homogeneity, Single Crystals, Growth Rate, Cooling Rate, Electrical Properties, Acceleration Effects, Rocket Motion, Vehicle Re-entry Forces/Vibration

Number of Samples: four
Sample Materials: Germanium doped with gallium. (The concentration of these samples ranged from 10^{17} to 10^{19} Ga atoms/cm^3.) (Ge*Ga*)
Container Materials: unknown

Experiment/Material Applications:
Improved crystalline materials are expected to result when samples are directionally solidified for extended periods of time in a reduced gravity environment. However, opportunities for such long-duration space flight solidification studies are limited. Because the low-gravity period available during a TEXUS sounding rocket flight is limited to only a few minutes, it was speculated that directional solidification studies might be inappropriate for such short flights. Thus, the prospect of obtaining suitable samples during sounding rocket flights was examined.

Some materials, such as germanium, can be directionally solidified at high growth rates and still yield high quality crystals. Such high growth rates had to be employed during the short-duration TEXUS flight to produce a significant amount of solidified crystal. Germanium proved to be an excellent sample material choice for the sounding rocket studies.

References/Applicable Publications:
(1) Walter, H. U.: Scientific Results and Accomplishments of the TEXUS Programme. In 33rd IAF International Astronautical Congress, Paris, France, September 27-October 2, 1982, p. 15, Paper No. #82-151. (post-flight)

(2) Walter, H. U.: Directional Solidification of Doped Semiconductors. In 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, Abstracts, ESA SP-191, p. 101. (post-flight)

(3) Hurle, D.T.J., Müller, G., and Nitsche, R.: Crystal Growth from the Melt. Chapter 10, in Fluid Sciences and Materials Science in Space, ESA, Springer-Verlag, Berlin, H. U. Walter, ed., p. 343. (post-flight)

(4) Directional Solidification of Doped Germanium. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 166-167. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 6
Launch Date/Expt Date: May 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 03
Builder of Processing Facility: Deutsche Forschungs-und Versuchsanstalt für Luft-und Raumfahrt (DFVLR), Federal Republic of Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft-und Raumfahrt (DLR).>

Experiment:
Dotation Stripes in Germanium (Ge)-Directional Solidification of Doped Semiconductors (two experiments designated as Experiment 1 and Experiment 2)

<Note: Walter performed two experiments during this TEXUS 6 mission. However, none of the available publications detailed the major differences between the two experiments. Therefore, the following summary describes the objectives, experimental setup and results from both investigations.>

This set of two TEXUS 6 experiments was the second in a series of investigations designed by Walter et al. to study the directional solidification of germanium doped with gallium (see Walter, TEXUS 4). The specific objective of the experiment was to investigate the feasibility of conducting simple crystal growth experiments during a sounding rocket flight.

Prior to the flight, two germanium samples were prepared. Each of the samples was doped with gallium: (1) concentration of 5×10^{17} Ga atoms/cm^3 and (2) concentration of 5×10^{18} Ga atoms/cm^3. Each sample was placed in a boron nitride crucible. Each of these crucibles was encapsulated in a tantalum cartridge.

The experimental procedure was the same as that employed during Walter's earlier TEXUS 4 experiments except that the samples were subjected to current pulsing: interface demarcations were placed in the samples by the application of a 50 ms current pulse every 4 seconds. The pulsing made it possible to (1) determine the interface shape and (2) quantify the microscopic growth rates.

Post-flight analysis of the single crystal samples (longitudinal sections) revealed "...an excellent homogeneity in the 2 cm long section corresponding to the 6 minutes of microgravity conditions. Striations were observed in the segments grown on the ground (gravity driven convection) and in the section corresponding to the despin of the rocket (forced convection)." (4, p. 168) Reportedly, it was determined that the lower-limit acceleration sensitivity of this type of crystal growth system is about 10^{-3} to 10^{-2} g which was higher than expected.

Further information concerning the specific analysis of each sample could not be located at this time.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Steady-State Solidification, Thermal Gradient, Semiconductors, Electronic Materials, Binary Systems, Solid/Liquid Interface, Interface Shapes, Interface Demarcation, Growth Rate, Dopant, Striations, Composition Variation, Sample Homogeneity, Single Crystals, Electrical Properties, Buoyancy-Driven Convection, Rocket Motion, Rotation of Payload, Rotating Fluids, Acceleration Effects

Number of Samples: two
Sample Materials: Germanium doped with gallium. Sample 1 had a concentration of 5 x 10^{17} Ga atoms/cm^3; Sample 2 had a concentration of 5 x 10^{18} Ga atoms/cm^3.
(Ge*Ga*)
Container Materials: Boron nitride crucible within a tantalum cartridge.
(B*N*, Ta*)

Experiment/Material Applications:
See Walter, TEXUS 4.

References/Applicable Publications:
(1) Walter, H. U.: Scientific Results and Accomplishments of the TEXUS Programme. In 33rd IAF International Astronautical Congress, Paris, France, September 27-October 2, 1982, p. 15, Paper No. #82-151. (post-flight)

(2) Walter, H. U.: Directional Solidification of Doped Semiconductors. In 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, Abstracts, ESA SP-191, p. 101. (post-flight)

(3) Hurle, D.T.J., Müller, G., and Nitsche, R.: Crystal Growth from the Melt. Chapter 10, in Fluid Sciences and Materials Science in Space, ESA, Springer-Verlag, Berlin, H. U. Walter, ed., p. 343. (post-flight)

(4) Striations in Germanium. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 168. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-2: Monoellipsoidal Mirror Furnace (ELLI)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Floating Zone Growth of Silicon

During crystal growth from a melt, convective flows within the molten material affect the impurity segregation at the solid-liquid interface. This impurity segregation can lead to compositional inhomogeneities which appear as striations in a doped crystal. The dopant distribution reflects the flow patterns which prevailed during crystal growth. The flow mechanisms, which are both dependent and independent of gravity, are superimposed on each other and difficult to discern during processing. During experiments performed in a low-g environment, gravity-driven flows are greatly reduced and contribution from the governing flow mechanisms can be distinguished.

This TEXUS 7 experiment was the first of a series of investigations designed by Eyer and/or Nitsche and/or Cröll et al. to study the flow mechanism responsible for striations observed in float-zone solidified silicon. The specific objectives of the research were to (1) compare the dopant distribution of crystals grown in space with those grown on Earth, and (2) demonstrate that float-zone crystal growth is feasible under low-gravity conditions.

Prior to the flight, a single phosphorus-doped silicon rod sample (length = 90 mm, diameter = 8 mm) with a [111] orientation was prepared. The rod was contained within a quartz ampoule (18 mm internal diameter) such that the sample material and ampoule wall were not touching. The ampoule was sealed and pressurized with argon to 760 Torr.

The sample was placed in the TEXUS Experiment Module TEM 02-2 ELLI Monoellipsoid Furnace. The module was configured with (1) a halogen lamp, (2) a monoellipsoidal mirror (a = 90 mm, b = 80 mm), (3) a sample translation and rotation mechanism, (4) a TV camera (which allowed observation and subsequent control of the process during the rocket flight), (5) experiment controllers and power supply, and (6) data transmission equipment. <Note: Eyer et al. had studied the low-gravity performance of halogen lamps (see Eyer, TEXUS 3, TEXUS 3b (Chapter 18)).>

Prior to rocket lift-off, the furnace was preheated and the sample was rotated at 10 rpm. During lift-off and rocket acceleration, the lamp was switched off to prevent filament damage. Once low-gravity conditions were achieved (65 seconds after lift-off), (1) the lamp was switched on and (2) a zone of the sample was melted while rotation continued. At 220 seconds after lift-off, zone translation was initiated at a rate of 5 mm/min. At 300 seconds after lift-off, sample rotation was terminated but zone translation continued. At 410 seconds (the beginning of rocket re-entry), the furnace power was switched off.

It was reported that the intended control of the experiment from the ground was not possible since "Up to T=250 [seconds] the TV picture was blurred. A hose, connecting TV camera and power supply, became leaky because it touched the hot rocket wall. The ensuing pressure drop caused corona discharges in the system, which later on, with the vacuum improving, disappeared." (1, p. 176)

Post-flight examination of the film and sample revealed that the zone length unexpectedly increased in length. The zone became so long that at t = 330 seconds zone instability and disruption occurred. "A tiny part of the melt was drawn to the lower interface and solidified there immediately. The major part collected at the upper interface forming a 'sphere' (diameter about 12 mm) of molten silicon." (1, p. 177) The increase in zone length was attributed to an increase in temperature of the sample. This temperature increase was attributed to the lack of convective cooling in the reduced gravity environment. Despite these difficulties, a sample length of about 10 mm was attained.

Examination of the flight and 1-g processed specimens revealed micro-striations present in both samples. It was, therefore, concluded that these striations were due to dopant inhomogeneities caused by oscillatory and turbulent modes of thermocapillary flow in the melt (see Reference (1) for a complete discussion of the results).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Float Zone Stability, Sample Rotation, Rotating Fluids, Dopant, Binary Systems, Segregation, Free Surface, Surface Tension, Surface Tension-Driven Convection, Thermocapillary Flow, Thermocapillary Convection, Marangoni Convection, Oscillatory Marangoni Convection, Turbulent Flow, Time Dependent Thermocapillary Flow, Solid/Liquid Interface, Sample Homogeneity, Impurities, Composition Variation, Rotational Striations, Striations, Buoyancy-Driven Convection, Absence of Convection (Detrimental), Translation Rate, Volume Change, Volume Expansion, Sample Deformation, Halogen Lamps

Number of Samples: one
Sample Materials: phosphorus doped silicon rod
(Si*P*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
See Experiment summary (above) and Cröll, TEXUS 12.

References/Applicable Publications:
(1) Eyer, A., Leiste, H., and Nitsche, R.: Floating Zone Growth of Silicon under Microgravity in a Sounding Rocket. Journal of Crystal Growth, Vol. 71, 1985, pp. 173-182. (post-flight)

(2) Floating-Zone Growth of a Silicon Single Crystal. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 174-175. (post-flight)

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Co-Investigator(s): Unknown
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Experiment Origin: France
Mission: TEXUS 7
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEM-SSC (Swedish Space Corporation) biellipsoidal mirror furnace
Builder of Processing Facility: Swedish Space Corporation, Tritonvägen, Solna, Sweden

Experiment:
Crystal Growth of Rare Earth - Magnesium Crystals

Several factors are involved in the growth of intermetallic compounds, including (1) thermodynamics of the system (enthalpies of formation, congruent melting) and (2) dynamic effects in the crystallization process (convection, diffusion, solidification-front shape). The dynamic effects, some of which are gravity-dependent, may prevent the crystallization of a congruent melting system. For instance, on Earth, sedimentation of heavier rare-Earth elements is detrimental to the formation of intermetallics.

This TEXUS 7 experiment was the first in a series of investigations designed by Pierre to study the growth of rare-Earth-magnesium alloys under low-gravity conditions. The specific objective of the experiment was to grow large crystals of the $CeMg_3$ intermetallic without the inhibiting effects of gravity.

During the mission, one Ce-Mg sample was processed in an biellipsoidal mirror furnace facility. The sample was heated to 800 ^{o}C and was molten for about 4 minutes of the 6-minute low-gravity period. It was reported that "...due to the geometry of this furnace, the temperature in the sample is rather inhomogeneous, which prevents the obtention [sic] of large single crystals." (1, p. 314) A reference sample was processed on Earth using similar experimental parameters.

Post-flight examination of the low-gravity processed sample revealed complete formation of the $CeMg_3$ phase. In contrast, the ground-processed sample also contained CeMg impurities. Reportedly, these results indicated that the segregation of Ce observed on Earth was due to density differences between the components rather than diffusion of atoms under the influence of a thermal gradient.

No further discussion of the results from this mission could be located in available publications.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Thermal Gradient, Rare Earth Alloys, Intermetallics, Binary Systems, Single Crystals, Solid/Liquid Interface, Solidification Front Physics, Interface Shapes, Density Difference, Segregation, Impurities, Convection, Diffusion, Sedimentation

Number of Samples: one
Sample Materials: cerium-magnesium compound
(Ce*Mg*)
Container Materials: tantalum
(Ta*)

Experiment/Material Applications:
The specific reason why $CeMg_3$ was selected for this experiment was not detailed in available publications.

References/Applicable Publications:
(1) Pierre, J., Baruchel, J., Schlenker, M., Siaud, E., Jönsson, R., and Holm, P.: Growth of $CeMg_3$ Crystals Under Microgravity. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987, pp. 313-316. (post-flight)

(2) Single Crystals of $CeMg_3$. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 170. (post-flight)

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Experiment Origin: France
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment, Experiment Module SSC
Processing Facility: Single lamp crystal growth furnace with pull capability
Builder of Processing Facility: Swedish Space Corporation, Trittonvägen, Solna, Sweden

Experiment:
Crystal Growth of Rare Earth Magnesium Crystals

This TEXUS 12 experiment was the second in a series of investigations designed by Pierre to study the growth of rare-Earth-magnesium alloys under low-gravity conditions (see Pierre, TEXUS 7). The specific objective of the experiment was to grow large crystals of $CeMg_3$ without the inhibiting effects of gravity.

During the mission, one sample of the stoichiometric compound $CeMg_3$ was processed in the Swedish Space Corporation image furnace. <Note: Reference (4), p. 52, states two samples were processed, whereas, Reference (2), pp. 57-59, refers to only one sample.> The material was contained in a sealed tantalum cartridge. A thermocouple was located in the conically shaped bottom of the cartridge.

During the low-gravity phase of the mission, the sample was heated to 820 ^{o}C and translated for about 20 mm, which allowed crystal growth. The power to the furnace was decreased prior to rocket re-entry allowing solidification. A similar sample was processed on Earth using the same thermal profile as the low-gravity processed sample.

Post-flight examination of the space-processed sample (metallography, x-ray back scattering for the surface, and neutron diffraction and topographic methods for the bulk) revealed platelet-shaped precipitates in the conical section of the sample (where crystallization started). These precipitates exhibited more or less coherent orientations over a large scale with domains of approximately 0.5 mm. In a similarly processed Earth-grown crystal, the precipitates were more randomly distributed with a polygonal cell shape and approximately 0.1 to 0.2 mm in size. The coherency of the space-grown crystal was greater

than the Earth-grown sample which was attributed to the lack of convection under low-gravity conditions.

It was further reported that the TEXUS 12 sample exhibited a restricted mosaicity while "...the Earth grown sample yielded X-ray patterns of worse quality." (3, p. 172) The neutron transmission Laue patterns showed a coherent of the flight sample while the 1-g processed material contained crystals of varying orientation. Rocking curve and topography studies of the low-gravity sample indicated that it consisted of two crystals disoriented by about 10^{o}. The internal mosaicity of each crystal was reported to be approximately 4^{o}. This result was attributed to the nucleation of several crystals in the conical end of the low-gravity sample.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Rare Earth Alloys, Intermetallics, Binary Systems, Single Crystals, Solid/Liquid Interface, Translation Rate, Solidification Front Physics, Interface Shapes, Surface Morphology, Nucleation, Platelet Habit, Density Difference, Segregation, Diffusion, Sedimentation, Precipitation, Buoyancy Effects Diminished, Lamps

Number of Samples: one
Sample Materials: Stoichiometric cerium-magnesium compound, $CeMg_3$ (Ce*Mg*)
Container Materials: tantalum
(Ta*)

Experiment/Material Applications:
The specific reason why $CeMg_3$ was selected for this experiment was not detailed in available publications.

References/Applicable Publications:
(1) Pierre, J., Baruchel, J., Schlenker, M., Siaud, E., Jönsson, R., and Holm, P.: Growth of $CeMg_3$ Crystals Under Microgravity. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987, pp. 313-316. (post-flight)

(2) Pierre, J. and Siaud, E.: Crystal Groth [sic] of Rare Earth-Magnesium Crystals. In TEXUS 11/12 Abschlussbericht 1985, BMFT/DFVLR, pp. 57-59. (post-flight)

(3) Growth of Rare Earth-Magnesium Crystals. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 172-173. (post-flight)

(4) Experiment-Modul SSC. In TEXUS 11/12 Abschlussbericht 1985, BMFT/DFVLR, p. 52. (post-flight; in German)

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: radiation from halogen lamps is focused on sample by two ellipsoidal mirrors
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Crystallization of a Silicon Drop 1ES324(A and B) A: Solidification with Rotation, B: Solidification Without Rotation

Time-dependent flow patterns in molten liquid silicon produce striations or doping inhomogeneities in the material. It is not clear if the sources of these flow patterns are (1) gravity-dependent buoyancy effects or (2) gravity-independent Marangoni effects (surface-driven flow caused by local differences in surface tension). In space, the gravity-dependent effects should be reduced allowing closer examination of Marangoni flows in the liquid silicon system.

This Spacelab 1 experiment was the first in a series of investigations designed by Kölker to study the solidification of a silicon drop under low-gravity conditions. The specific objective of the investigation was to determine if surface-driven flow exists in liquid silicon.

During the Spacelab 1 mission, the Mirror Heating Facility (MHF) was used to melt and directionally solidify a portion of an n-type doped Si rod (Czochralski-grown on Earth, [100] orientation). The 11 mm diameter, 50 mm long rod was mounted in the MHF and melted at its free end so that a drop (1 cm^3) of liquid silicon formed. The sample was rotated at 10 rpm for 10 minutes and then slowly withdrawn from the hot portion of the furnace at 1 mm/min. <Note: It is unclear if rotation continued during withdrawal.> This processing of the sample resulted in the directional solidification of the silicon. It was reported that, while molten, the silicon drop was spherical. "Because of a certain peculiarity of the wetting angle... the growing crystal very strongly deviate[d] from the spherical shape and assume[d] more or less the shape of a rocket nose [once solidified]." (1,

p. 170)

A second experimental run was planned during which the sample would be processed without rotation. Reportedly, this second procedure "...could not be carried out because of breakdown of the furnace." (1, p. 169)

Post-flight examination of the single, resolidified Spacelab sample indicated that a thin, dark surface layer had collected on the specimen. It was suspected that this layer was due to a carbon impurity of an unknown origin. Reportedly, once the droplet was etched, this layer was removed and the sample was very shiny.

After longitudinally slicing, polishing and etching the sample (see Reference (1) for procedure), distinct rotational striations were revealed at the surface. It was expected that the distance between neighboring striation lines would be about 50 microns. However, distances varied considerably (from 40 to 300 microns). This variation in line distance was attributed to a variation in growth rate caused by the carbon impurity. Because the melting point of Si varies greatly with small changes in carbon content, "So the growth rate rises with increasing carbon content and must fall back to the value of 1 mm/min imposed by the driving motor when the carbon content is held constant by precipitation of Si-C inclusions." (1, p. 170)

Less distinct non-rotational striations were found between the more distinct rotational striations described above and were spaced at a distance of about 10 microns. Because of the presence of these non-rotational striations, it was assumed that "...other temperature fluctuations in addition to the rotation in the twofold symmetry [existed]. In the light of our earth bound experiments it is very tempting to assume that they are indeed caused by the Marangoni effect." (1, p. 171) Reportedly, to confirm this assumption, the planned (but not realized) experiment without rotation would have been helpful.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Containerless Processing, Binary Systems, Semiconductors, Semiconductor Applications, Electronic Materials, Dopant, Drops, Spheres, Sphericity, Sample Rotation, Drop Rotation, Rotating Fluids, Growth Rate, Translation Rate, Free Surface Solidification, Solid/Liquid Interface, Liquid/Gas Interface, Free Surface, Surface Tension, Surface Tension-Driven Convection, Thermocapillary Flow, Marangoni Convection, Time Dependent Thermocapillary Flow, Wetting, Contact Angle, Sample Deformation, Surface Morphology, Striations, Precipitation, Inclusions, Rotational Striations, Sample Homogeneity, Contamination Source, Impurities, Thin Films, Coated Surfaces, Halogen Lamps, Furnace Malfunction

Number of Samples: one
Sample Materials: silicon rod, n-type doped, [100] orientation (Si*)
Container Materials: not applicable

Experiment/Material Applications:
Silicon is a material widely used for electronic applications. Doping inhomogeneities (striations) are detrimental to the quality of electronic devices which are fabricated from silicon.

References/Applicable Publications:
(1) Kölker, H.: Crystallization Of a Silicon Sphere. In ESA 5th European Symposium on Material Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 169-172. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. Teledyne Brown Engineering publication (acceleration measurements on Spacelab 1)

(3) Nitsche, R.: The Mirror Heating Facility: Performance and Experiences. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November, 5-7, 1984, ESA SP-222, pp. 155-156. (post-flight; discusses experimental hardware)

(4) Sanz, A.: The Crystallization of a Molten Sphere. Journal of Crystal Growth, 74 (1986), pp. 642-655. (calculations of molten sphere crystallization as compared to Kolker results; post-flight)

(5) Sanz, A., Meseguer, J., and Mayo, L.: The Influence of Gravity on the Solidification of a Drop. Journal of Crystal Growth, 82 (1987), pp. 81-88. (numerical calculations of molten sphere solidification as compared to Kolker results; post-flight)

(6) Eyer, A., Nitsche, R., and Zimmermann, H.: A Double-Ellipsoid Mirror Furnace for Zone Crystallization Experiments in Spacelab. Journal of Crystal Growth, 47 (1979), pp. 219-229. (furnace setup)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: radiation from two halogen lamps is focused on sample by two ellipsoidal mirrors
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Crystallization of a Silicon Sphere (A study of the low "g" effects on convective flow and crystal perfection) (WL-MHF-04)

This Spacelab D1 experiment was the second in a series of investigations designed by Kölker to study the solidification of a silicon drop under low-gravity conditions (see Kölker, Spacelab 1).

During the earlier Spacelab 1 mission, a silicon drop was processed while subjected to rotation. Post-flight evaluation of the processed drop indicated that not only rotational striations were present in the sample but non-rotational striations as well. It was assumed that these non-rotational striations were caused by time-dependent, surface-driven flows. Although a similar experiment performed without rotation would have helped prove these assumptions, the planned Spacelab 1, non-rotation experiment could not be performed because of a furnace malfunction. Therefore, the objectives of this Spacelab D1 investigation were to determine (1) if similar striations result when the sample is not rotated and (2) if surface-driven flow exists in the liquid silicon.

During the Spacelab D1 mission, the Mirror Heating Facility was used to melt and directionally solidify an n-type doped, [100] oriented, silicon sample. The experimental procedure was the same as that used during the Spacelab 1 mission (see Kölker, Spacelab 1) except that the sample was not rotated.

Post-flight examination of the longitudinally sliced, polished, and etched sample revealed a dense pattern of striations. The microscopic growth rate at various parts of the sample (indicated

by the separation distance of striations) fluctuated greatly. This result indicated the presence of time-dependent flow in the melt. The fluctuations were sometimes periodic. After approximately two-thirds of the melt had solidified, the time-dependent flow was less pronounced. However, near the end of the sample, it increased. Comparison of these results to those of samples processed on Earth from sub-critical Rayleigh melts (containing samples of a height less than 1 cm) indicated that the non-rotational striations were due to surface-driven flows.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Thermal Gradient, Containerless Processing, Binary Systems, Semiconductors, Semiconductor Applications, Electronic Materials, Dopant, Drops, Spheres, Sphericity, Growth Rate, Free Surface Solidification, Solid/Liquid Interface, Liquid/Gas Interface, Free Surface, Surface Tension, Surface Tension-Driven Convection, Thermocapillary Flow, Marangoni Convection, Time Dependent Thermocapillary Flow, Surface Morphology, Striations, Sample Homogeneity, Halogen Lamps

Number of Samples: one
Sample Materials: silicon rod, n-type doped, [100] orientation (Si*)
Container Materials: not applicable

Experiment/Material Applications:
See Kölker, Spacelab 1.

References/Applicable Publications:
(1) Kölker, H., and Nitsche, R.: Crystallization of a Silicon Sphere. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 111-112. (preflight)

(2) Kölker, H.: Crystallization of a Silicon Sphere. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 264-267. (post-flight)

(3) Sanz, A., Meseguer, J., and Mayo, L.: The Influence of Gravity on the Solidification of a Drop. Journal of Crystal Growth, 82 (1987), pp. 81-88. (numerical simulations of molten sphere solidification, related to Kolker research; post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: Radiation from halogen lamps is focused on sample by two ellipsoidal mirrors
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Floating Zone Growth of Silicon (1ES321)

This Spacelab 1 experiment was the second in a series of investigations designed Nitsche and/or Eyer and/or Cröll et al. to study the flow mechanism responsible for striations observed in float-zone solidified silicon (see Eyer, TEXUS 7 (this chapter)). The specific objective of the experiment was to determine if such striations were due to gravity-dependent flow (thermosolutal convection) or gravity-independent flow (thermocapillary Marangoni convection).

Prior to the shuttle flight, two phosphorus-doped (6×10^{18}/cc), silicon rods (10-mm diameter, 112-mm long) were prepared for the experiment. Since the furnace, rather than the sample, was to be translated during the float-zone processing (for space and weight savings), the rods had prefabricated necks.

Two different float-zone experiments were attempted during the mission. Each of these experiments had the same processing procedure. During the first experiment, one of the samples was inserted into the Spacelab Mirror Heating Facility (MHF) furnace. The furnace was (1) evacuated and back-filled with argon (900 Torr) three times and then (2) switched on. Once a molten zone was established in the lower portion of the neck, (1) a 5 mm/min furnace translation was initiated and (2) the sample was rotated. Once the zone passed through the neck region, rotation was halted and the zone translated through the thicker sample section. After a certain growth period, rotation was again initiated and continued until the end of the experiment. The entire process was filmed, which allowed observation and control of zone stability (by heater power adjustment) by the Payload Specialist.

Reportedly, processing of the Si sample caused an irregular, wavy neck shape. This wavy shape illustrated zone stability control difficulties.

Reportedly, "The second experiment had to be terminated shortly after starting zone travel because of disrupture of the melt. Apparently the zone length had become too large and the crystal diameter had decreased to such a degree that disruption near the lower interface occurred." (1, p. 177) <Note: Additional details describing the performance of the MHF can be found in Reference (6).>

Post-flight examination of the completely processed Si sample (from the first experiment) was performed using microscopic techniques, microphotography, and the assemblage of "striation topograms." The following conclusions were reported:

"-It could be shown experimentally, by elimination of gravity driven and electromagnetically induced flows, that time dependent thermocapillary flows are the cause for microstriations in silicon crystals float zoned in space.

"-The similarity between the microstriation patterns of flight and reference [lg processed] crystal[sic] suggests that the cause of microstriations on earth is also time dependent thermocapillary flow. Float zoned crystals, during the growth of which the melt was covered with a coating, showed no striations. <Note: It is not clear if the author is referring to ground-based or low-gravity coated float-zone crystal growth.>

"-The fact that the distances between the striations are not periodic, but irregular, is probably due to the existence of turbulent thermocapillary flows under the extreme conditions in silicon melts.

"-Zone forms and interface shapes are different on earth and in space.

"-Zone stability under low gravity can be problematic. The transient decrease in crystal diameter, caused by the melt, trying to maintain a meniscus angle of 11 [degrees], can lead to zone disruption." (1, p. 180)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Coated Float Zones, Float Zone Stability, Liquid Column Rupture, Single Crystals, Sample Rotation, Dopant, Binary Systems, Segregation, Free Surface, Surface Tension, Surface Tension-Driven Convection, Thermocapillary Flow, Thermocapillary Convection, Marangoni Convection, Oscillatory Marangoni Convection, Thermosolutal Convection, Turbulent Flow, Time Dependent Thermocapillary Flow, Translation Rate, Solid/Liquid Interface, Liquid/Gas Interface, Interface Shapes, Interfacial Curvature, Meniscus Shape, Sample Homogeneity, Composition Variation, Striations, Sample Deformation, Halogen Lamps

Number of Samples: two
Sample Materials: silicon doped with phosphorous (6 x 10^{18}/cc); processing atmosphere: argon
(Si*P*, Ar*)
Container Materials: not applicable

Experiment/Material Applications:
See, Eyer, TEXUS 7.

References/Applicable Publications:
(1) Eyer, A., Leiste, H., and Nitsche, R.: Crystal Growth Of Silicon On Spacelab 1, Experiment ES-321. In ESA European Symposium on Material Science Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 173-182. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(3) Martinez, I. and Eyer, A.: Liquid Bridge Analysis of Silicon Crystal Growth Experiments Under Microgravity. Journal of Crystal Growth, 75 (1986), pp. 535-544. (post-flight; math analysis)

(4) Eyer, A., Nitsche, R., and Zimmerman, H.: A Double-Ellipsoid Mirror Furnace for Zone Crystallization Experiments on Spacelab. Journal of Crystal Growth, 47 (1979), p. 219. (furnace setup)

(5) Eyer, A. and Leiste, H.: Growth of Silicon Single Crystal by the Floating-Zone Technique Under Low-gravity Conditions During Spacelab-1. Final Report, Bundesministerium für Forschung und Technologie, BMFT-FB-W 86-004, January 1986. (post-flight; English summary)

(6) Nitsche, R.: The Mirror Heating Facility: Performance and Experiences. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 155-156. (post-flight)

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Co-Investigator(s): None
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<Note: The Principal Investigator listed a new address in Garches, France. However, the name of the present company/university that employs the Investigator was not indicated.>

Experiment Origin: France
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility
Processing Facility: Gradient Heating Facility (GHF) Furnace
Builder of Processing Facility: CNES, France

Experiment:
Lead Telluride Crystal Growth (1ES318)

<Note: Very little information concerning this experiment was published in English. References (1), (4), and (5) were not translated prior to the preparation of this experiment summary. It was, therefore, difficult to discern the exact experimental objectives, setup and procedures of the experiment. The following summary was based on Reference (1) (a NASA RECON abstract (published in English)) and Reference (3).>

During this STS-009 experiment, three PbTe monocrystals were grown by the Bridgman method in the Spacelab Gradient Heating Facility (GHF). (Two crystals were grown from seeds and one was grown without a seed.) Reportedly, the samples were directionally solidified at a rate of 1 cm/h by lowering the furnace temperature (producing a 25 ^{o}C/cm thermal gradient on the samples).

The low-gravity samples were compared to two samples processed on Earth under similar thermal conditions. Examination of all samples revealed a significantly lower dislocation density in the space-processed samples (10^{-4} and 10^{-3} cm^{-2}) when compared to the 1-g materials (>10^{-4} cm^{-2}).

<Note: Although the Principal Investigator briefly reported that an silver (Ag) impurity segregation was investigated, no further comments were made (in English) concerning the results of this specific analysis. The investigator did indicate that these results could be found in the French publication, Reference (4), p. 81, figure 4.>

Reportedly, the results from this experiment revealed that it is possible to grow large (17 mm diameter) crystals in space which are of better quality than crystals produced on Earth. However, it was noted that "the rate and stability of accelerations" are the main factors affecting successful, low-gravity crystal growth. Reportedly, these factors exclude using manned vehicles or spacecraft with multiple payloads incompatible with crystal growth requirements. <Note: A further discussion of these accelerations/factors was not available in English.>

No other information concerning this experiment (published in English) could be located at this time.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Bridgman Technique, Thermal Gradient, Binary Systems, Single Crystals, Seed Crystals, Growth Rate, Solid/Liquid Interface, Sample Homogeneity, Dislocations, Contamination Source, Impurities, Infrared Detector Applications, Acceleration Effects

Number of Samples: three
Sample Materials: lead telluride
(Pb*Te*)
Container Materials: The samples were sealed in silica ampoules.

Experiment/Material Applications:
PbTe materials are used in infrared detection devices.

This experiment was performed to determine if the low-gravity processed PbTe crystals were of superior quality over 1-g processed PbTe crystals.

References/Applicable Publications:
(1) Rodot, H. and Totereau, O.: Lead Telluride Crystals Grown In Microgravity Experiment ES 318. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 135-139. (post-flight; in French; English abstract obtained from NASA RECON)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab 1)

(3) Input received from Experiment Investigator, October 1989 and 1993.

(4) Rodot, H., Regel, L. L., Sarafanov, G. V., Hamidi, M., Videskii, I. V., and Turtchaninov, A. M.: Cristaux de Tellurure de Plomb Élaborés en Centrifugeuse. J. of Crystal Growth, 79 (1986), pp. 77-83. (in French)

(5) Rodot, H.: Croissance de Cristaux de Tellurure de Plomb en Microgravite. Document sent by H. Rodot to J. Jones (Wyle), August 1993. (in French)

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Experiment Origin: Sweden
Mission: TEXUS 10
Launch Date/Expt. Date: May 1984
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Microfurnaces employing parabolic reflectors
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Floating Zone Growth of Ge Crystals from a Melt Zone

In the reduced gravity environment of a sounding rocket, the overwhelming effects of buoyancy-driven convection are greatly reduced, allowing a closer examination of surface tension forces and their critical role in driving convective thermal and mass transport within a floating zone.

This TEXUS 10 experiment was the second in a series of investigations designed by Carlberg et al. to study low-gravity float-zone crystal growth (see Carlberg, TEXUS 7, (Chapter 14)). The specific objective of the research was to assess the effect of Marangoni convection on crystal morphology by comparing the resultant dopant distributions of a germanium crystal processed in (1) a reduced gravity environment and (2) in an Earth-based laboratory.

Prior to the rocket launch, two samples of gallium doped germanium were placed within mirror furnaces. Reportedly, the furnaces employed parabolic reflectors. "The parabolic shape of the reflectors concentrates the light from the lamp to a focus in the sample." (1, p. 367)

During the low-gravity phase of the rocket, a floating zone was created in each of the rod-shaped, 5 mm diameter Ge-Ga single crystals. Then the samples were "...pulled at 3 mm/mn on 5 mm and 5 mm/mn on 7 mm respectively. Current pulsing with a frequency of 1 Hz was applied for the marking of the growing solid/liquid interface." (6, p. 180)

Reportedly, "The growth rates along the [flight] samples could be deduced from the interface demarcations and segregation profiles could be measured. These profiles clearly showed that convective mixing... [occurred] in the liquid zones. But the absence of

striations indicated that the driving force of the convection was not strong enough to generate oscillatory flows." (6, p. 180)

Dopant homogeneity of the space-processed crystals was compared to dopant homogeneity of Earth-processed crystals. Reportedly, "...convective mixing occurred both in space and on earth. Fluid dynamic analysis showed that Marangoni convection was the origin of the mixing in the space samples. Results from samples processed on earth indicate a somewhat stronger convection." (1, p. 367) Further, "The similarities observed between the convection levels in the space samples and in reference samples showed the importance of the contribution of surface tension forces to the convection flows in liquid zones on the ground." (6, p. 180)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Binary Systems, Single Crystals, Dopant, Surface Tension, Free Surface, Heat and Mass Transfer, Surface Tension-Driven Convection, Thermocapillary Convection, Marangoni Convection, Oscillatory Marangoni Convection, Buoyancy-Driven Convection, Growth Rate, Pulling Rate, Solid/Liquid Interface, Liquid/Gas Interface, Segregation, Striations, Surface Morphology, Sample Homogeneity, Interface Demarcation, Lamps

Number of Samples: two
Sample Materials: gallium-doped germanium (Ge*Ga*)
Container Materials: unknown

Experiment/Material Applications:
See Carlberg, TEXUS 7.

References/Applicable Publications:
(1) Carlberg, T.: A Preliminary Report on Floating-Zone Experiments with Germanium Crystals in a Sounding Rocket. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 367-373. (post-flight)

(2) Carlberg, T. and Liljendahl, M.: Crystal Growth Furnace Developed for Floating Zone Experiments in Sounding Rockets. In Proceedings of the 7th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Loen, Norway, May 5-11, 1985, ESA SP-229, July 1985, p. 165.

(3) Carlberg, T.: Floating Zone Experiments with Germanium Crystals in Sounding Rockets. 36th IAF International Astronautical Congress, Stockholm, Sweden, October 7-12, 1985, IAF Paper #85-272, 7 pp.

(4) Carlberg, T.: Lateral Solute Segregation during Floating-Zone Crystal Growth Under Different Gravity Conditions. Journal of Crystal Growth, 79 (1986), pp. 71-76.

(5) Input received from Experiment Investigator, May 1988.

(6) Floating Zone Crystal Growth of Ga-Doped Germanium. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 180-181. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Microfurnaces employing parabolic reflectors
Builder of Processing Facility: Swedish Space Corporation, Solna, Sweden

Experiment:
Floating Zone Experiments with Germanium Crystals

This TEXUS 12 experiment was the third in a series of investigations designed by Carlberg to study low-gravity float-zone crystal growth (see Carlberg, TEXUS 7 (Chapter 14), TEXUS 10 (this chapter)). The specific objective of the experiment was to investigate the effects of thermocapillary convection during the float-zone processing of coated gallium-doped germanium crystals.

In preparation for the experiment, 5 mm diameter Ga-doped Ge samples were housed within Pyrex™ tubes. The inner diameter of these tubes matched that of the samples (5 mm) while the outer diameter of the tubes was 7 mm.

The samples were processed within mirror furnaces employing parabolic reflectors. "During heating, the... [Pyrex™] glass (silica glass with a softening temperature of about 600 ^{o}C) collapses towards the sample, and when the liquid zone forms the melt wets the glass tube." (4, p. 1) Interface demarcations of the solid/liquid interface via current pulsing allowed an evaluation of the interface shape and growth rate of the samples. <Note: Reference (6) appears to indicate that both free surface and covered surface experiments were performed on this flight. However, no further description of the preparation of these free surface experiments was presented in the available references. Reference (6) may have been referring to earlier TEXUS 10 results. Further, the Principal Investigator indicated that a total of three samples were to be processed but that two "failed." Perhaps one or more of these two samples contained free surfaces.>

While TEXUS 10 experiments had indicated that uncoated Ga-doped Ge crystals had resulted in convective mixing similar to that of ground-processed uncoated samples, these TEXUS 12 results indi-

cated that the coated crystals (1) demonstrated decreased convection in the liquid zone and (2) exhibited an axial segregation which was "close to diffusion controlled." Further, analysis of radial segregation during floating zone crystal growth under several different experimental conditions (1-g coated, 1-g uncoated, low-g coated, and low-g uncoated) demonstrated that the systems had a high sensitivity to interface shape and to convection. The low-g coated sample exhibited an inverted radial concentration profile when compared to the others. "This can only be interpreted as the result of a very weak convection, which was not strong enough to... [homogenize] the melt but which caused a flow of solute from regions close to the surface towards the centre.

"For the three other samples, where the Ga concentration is higher close to the surface region, the results can be explained by outwards diffusion along the curved solid/liquid interface which occurred in the boundary layer while the bulk melt is assumed to have been homogeneous due to convective mixing." (6, p. 188)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Encapsulated Float Zones, Coated Float Zones, Coated Surfaces, Binary Systems, Dopant, Surface Tension, Thermocapillary Convection, Marangoni Convection, Marangoni Convection Diminished, Liquid/Liquid Interface, Free Surface, Liquid/Gas Interface, Solid/Liquid Interface, Solidification Front Physics, Interface Shapes, Wetting, Growth Rate, Interface Demarcation, Homogeneity, Segregation, Axial Segregation, Diffusion-Controlled Growth, Semiconductor Applications, Lamps, Sample Not Processed as Planned

Number of Samples: three
Sample Materials: Germanium (crystal), 5 x 10^{18} at/cm^3 gallium (dopant). The samples were processed in an argon atmosphere.
(Ge*Ga*)
Container Materials: encapsulating material: Pyrex^{TM}; container material: quartz
(Si*O*)

Experiment/Material Applications:
Space float zone processing may lead to the improvement of the homogeneity, purity, and defect distribution of semiconductor crystals.

References/Applicable Publications:
(1) Carlberg, T.: Floating Zone Experiments with Germanium Crystals. In TEXUS 11/12 Abschlussbericht 1985, German publication, pp. 53-54. (post-flight)

(2) Carlberg, T.: Floating Zone Experiments with Germanium Crystals in Sounding Rockets. Acta Astronautica, Vol. 13, Nos. 11/12 (1986) pp. 639-643.

(3) Carlberg, T.: Lateral Solute Segregation During Floating Zone Crystal Growth Under Different Gravity Conditions. Journal of Crystal Growth, 79 (1986), pp. 71-76.

(4) Carlberg, T.: Floating Zone Experiments with Germanium Crystals in Sounding Rockets. 36th IAF International Astronautical Congress, Stockholm, Sweden, October, 7-12, 1985, IAF Paper #85-272, 7 pp. (post-flight)

(5) Input received from Experiment Investigator, May 1988.

(6) Floating-Zone Experiments with Germanium. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 188-189. (post-flight)

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Experiment Origin: Sweden
Mission: TEXUS 14a
Launch Date/Expt. Date: May 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation Experimental Module: Four crystal growth microfurnaces employing a halogen lamp and parabolic reflectors
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Floating Zone Experiments with Germanium Crystals

This TEXUS 14a experiment was the fourth in a series of investigations designed by Carlberg to study low-gravity float-zone crystal growth (see Carlberg, TEXUS 7 (Chapter 14), TEXUS 10, and TEXUS 12 (this chapter)).

Reportedly, due to an unexpected "wobbling motion" of the TEXUS 14a rocket, uncontrollable accelerations were produced on the vehicle and the desired low gravity level of 10^{-4} g was not attained. The experiment was reflown on TEXUS 14b. (See Carlberg, TEXUS 14b.)

Documentation detailing any results of this TEXUS 14a experiment does not appear to be available.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Surface Tension, Thermocapillary Convection, Marangoni Convection, Solid/Liquid Interface, Liquid/Gas Interface, Semiconductor Applications, Rocket Motion, Acceleration Effects

Number of Samples: unknown
Sample Materials: Unknown, probably gallium-doped germanium. (Ge*Ga*)
Container Materials: unknown

Experiment/Material Applications:
See Carlberg, TEXUS 12.

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 53-55. (in German; post-flight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht pp. 60-61. (crystal growth furnaces)

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Experiment Origin: Sweden
Mission: TEXUS 14b
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: ESA/Swedish Space Corporation Experimental Module: Four crystal growth microfurnaces employing a halogen lamp and parabolic reflectors
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Floating Zone Experiments with Germanium Crystals

This TEXUS 14b experiment was the fifth in a series of investigations designed by Carlberg et al. to study low-gravity float-zone crystal growth (see Carlberg, TEXUS 7 (Chapter 14), TEXUS 10, TEXUS 12, and TEXUS 14a (this chapter)). The specific objective of the experiment was "...to vary the length of the liquid zones and thereby also the shape of the growing interfaces... [longer zones exhibit more planar interfaces]." (1, p. 75)

The experiments were extensions of research initiated by Carlberg on TEXUS 10 and 12. During TEXUS 14b, both coated and uncoated samples of gallium-doped germanium crystals were processed. During the mission, two, 5 mm diameter, uncoated (or free surface) samples and two, 5 mm diameter, Pyrex™-coated samples were melted and solidified within four crystal growth furnaces in the ESA/Swedish Space Corporation Module. Current pulsing was applied to mark the growing solid/liquid interface. The sample processing sequence was programmed to produce a coated long zone (12-13 mm), a coated short zone (3-4 mm), an uncoated long zone (6 mm), and an uncoated short zone (3 mm).

"In the two experiments programmed to give short liquid zones... the liquid zones became too short prior to the end of the pulling sequence... these experiments terminated earlier than expected and produced relatively short sections of grown material.

"Of the two coated samples, the one grown with a short liquid zone... showed good wetting between the pyrex[sic] glass and the Ge crystal. The other one... had pores between the pyrex[sic] glass and the Ge from the start of growth to a point where about 8 mm of the crystal had been grown." (4, p. 62)

Reportedly, the two uncoated crystals exhibited similar results to the TEXUS 10 experiments. "Analysis of axial segregation shows strong evidence of [surface tension driven] convection in the liquid zones, and especially in the sample with a long liquid zone the value of the effective distribution coefficient is very low...." (1, p. 76) (Because the Marangoni number is directly proportional to zone length, it was expected that increased Marangoni convection would occur in the longer zones.) Further, "Radial segregation of solute occurred towards the periphery of both [uncoated] samples, thereby indicating a prevailing influence of the convex shape of the growing interface with regard to the convection pattern in the liquid zone." (5, p. 184)

Results obtained via analysis of the two coated samples were "more unexpected". Reportedly, "The axial concentration profile in the sample with [the] short zone corresponds to purely diffusive conditions." (5, p. 184) Further, "The axial dopant distribution in the covered sample with [the] long zone... exhibits an initial transient and a period of steady-state conditions. After that, the concentration [in the long zone] begins to rise... whereas the corresponding growth rate was slowly decreasing. Also weak striations (frequency about 1 Hz) are randomly distributed over about 8 mm from the growth start. As the pyrex[sic] was not well wetted by the liquid over these 8 mm length, these results could be explained by Marangoni convection flows occurring locally at the periphery of the sample." (5, p. 184)

A more detailed discussion of the analysis related to each sample was presented in Reference (4).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Encapsulated Float Zones, Coated Float Zones, Coated Surfaces, Float Zone Stability, Binary Systems, Dopant, Free Surface, Surface Tension, Surface Tension-Driven Convection, Thermocapillary Convection, Marangoni Convection, Liquid/Liquid Interface, Liquid/Gas Interface, Solid/Liquid Interface, Solidification Front Physics, Steady-State Solidification, Planar Solidification Interface, Interface Shapes, Wetting, Wetting of Container, Non-Wetting of Container, Growth Rate, Interface Demarcation, Homogeneity, Segregation, Axial Segregation, Pores, Striations, Diffusion-Controlled Growth, Semiconductor Applications, Halogen Lamps, Processing Difficulties

Number of Samples: four
Sample Materials: Germanium doped with 5 x 10^{18} gallium. The samples were processed in an Argon atmosphere. (Ge*Ga*)
Container Materials: encapsulation material: Pyrex^{TM}; quartz ampoules

Experiment/Material Applications:
Space float-zone processing may lead to the improvement of the homogeneity, purity and defect distribution of the semiconductor crystals.

References/Applicable Publications:
(1) Carlberg, T. and Tillberg, E.: Floating Zone Experiments with Germanium Crystals. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 75-78. (post-flight)

(2) Experiment-Module ESA/SSC. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 60-61. (crystal growth furnace description)

(3) Input received from Experiment Investigator, May 1988.

(4) Tillberg, E. and Carlberg, T.: The Influence of Convection on the Solute Distribution in Crystals Grown by Floating Zone Technique. Appl. Microgravity Tech. II (1989), 2, pp. 61-67. (post-flight)

(5) Floating Zone Growth of Germanium Crystals. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 184-185. (post-flight)

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Experiment Origin: Sweden
Mission: MASER 2
Launch Date/Expt. Date: February 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Crystal Growth Furnace (CGF) (The CGF Module contained four ellipsoidal mirror furnaces)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Semi-Confined Bridgman Growth of Ga Doped Ge

This MASER 2 experiment was the sixth in a series of investigations designed by Carlberg to study low-gravity floating zone crystal growth (see Carlberg, TEXUS 7 (Chapter 14), TEXUS 10, TEXUS 12, TEXUS 14a, and TEXUS 14b (this chapter)). The specific objectives of the experiment were to (1) investigate the semi-confined Bridgman growth of gallium-doped germanium, (2) study the effect of thermocapillary convection (originating at the free surfaces) on the dopant segregation in the crystals, and (3) determine (if possible) the critical Marangoni number corresponding to the onset of oscillatory flows.

Prior to the rocket launch, two, 6 mm diameter samples were prepared such that when each was processed, 14 mm of a 20 mm molten zone would be coated with quartz glass. Using two of the four ellipsoidal furnaces housed within the Crystal Growth Furnace (CGF), the two samples were melted and then directionally solidified during the mission. Current pulsing was applied to mark the growing liquid/solid interface. Sample mounting and heat shielding differences of the two samples allowed one of the samples to be processed with a thermal gradient of 130 K/cm and the other with a thermal gradient of 90 K/cm. A thermocouple, placed at the border between the coated and uncoated sections of the sample, was used to record this thermal gradient.

Reportedly, "The outprints of data from the flight, show temperature and power curves in fairly good agreement with the programmed sequences. It can be concluded that the two furnaces seem to have been functioning normally." (3, p. 6) Further, it was noted that "...an inspection of the surfaces shows that liquid zones of expected extensions (18-19mm) have been established in both samples." (3, p. 6)

"The axial segregation profile in the high gradient [low-g] sample... corresponded to an effective partition coefficient of 0.75 and indicated that only a weak convection occurred during the growth. In the low-gradient [low-g] sample, a strong coupling occurred between the unstable growth rate and the concentration as well as a strong radial segregation.... [The results from both samples were] contrary to expectations, since the Marangoni numbers were of the order of 10^4 when the growth started, and significant convection including oscillatory flows-should have occurred. This result could however be explained by the presence of a native oxide layer on the surface of the samples. The layer cracked on melting and only a small free surface area could generate Marangoni convection.

"As the growing interface became concave in the covered region, the radial segregation occurred towards the center of the samples.... The asymmetry of the radial segregation profiles was probably also due to the shape of the interface which was perturbed by the thermocouples located in the samples." (6, p. 186)

A related experiment can be found under Camel, MASER 2 (this chapter).

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Directional Solidification, Bridgman Technique, Semi-Confined Bridgman Growth, Encapsulated Float Zones, Coated Float Zones, Coated Surfaces, Oxide Layer, Float Zone Stability, Binary Systems, Dopant, Free Surface, Surface Tension, Thermocapillary Convection, Marangoni Convection, Oscillatory Marangoni Convection, Marangoni Convection Diminished, Liquid/Liquid Interface, Liquid/Gas Interface, Solid/Liquid Interface, Solidification Front Physics, Interface Shapes, Interface Curvature, Wetting, Thermal Gradient, Growth Rate, Interface Demarcation, Liquid Film Rupture, Homogeneity, Segregation, Axial Segregation, Striations, Semiconductor Applications, Lamps

Number of Samples: two
Sample Materials: germanium doped with gallium (Ge*Ga*)
Container Materials: quartz glass coating (Si*O*)

Experiment/Material Applications:
Space float-zone processing may lead to the improvement of the homogeneity, purity, and defect distribution of semiconductor crystals.

References/Applicable Publications:
(1) Zaar, J. and Änggard, K.: Maser and Its Effectiveness and Experimental Results. In: In Space '87, Japan Space Utilization Promotion Center (JSUP), October 13-14, 1987, 32 pp. (preflight)

(2) Zaar, J. and Dreier, L.: MASER II Final Report. RMLO/1-7 Swedish Space Corporation, August 30, 1988.

(3) Semi-Confined Bridgman Growth of Germanium Crystals. In MASER II Final Report, RMLO/1-7 Swedish Space Corporation, August 30, 1988, Appendix 5, p. 6. (post-flight)

(4) Input received from Experiment Investigator, May 1988.

(5) Input received from Experiment Investigator, October 1989.

(6) Semi-Confined Bridgman Growth of Ga-doped Ge. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 186-187. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #12, STS-014 (STS 41-D, Discovery)
Launch Date/Expt. Date: August 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment, Shuttle Student Involvement Program (SSIP), Middeck Experiment
Processing Facility: Fluids Experiment Apparatus (FEA): configured for float-zone processing and middeck installation
Builder of Processing Facility: Rockwell International, Downey, California

Experiment:
Float-Zone Single Crystal Growth of Indium (SE82-14)

This STS-014 experiment was designed to investigate the float-zone crystallization of thallium-doped indium. The specific objectives of the research included (1) determining the distribution coefficient of Tl in In and (2) examining the crystalline structure of the shuttle-processed sample.

Prior to the flight, a 16.2 cm section of polycrystalline indium doped with 100 ppm thallium was welded to a 5.4 cm section of an indium single crystal. The welded sample was then enclosed within a sealed glass tube, filled with argon (pressure: 1 atm), and installed into the Fluids Experiment Apparatus (FEA).

At the beginning of the space experiment, a 2-cm long area at the interface of the polycrystalline and single crystal was melted by an electrical resistance heater which surrounded the glass tube. The heater then translated at a rate of 2.5 cm/hr in the direction of the polycrystalline.

Reportedly, because the experiment initiation took longer than anticipated and some unexpected hardware difficulties arose, the actual sample processing time was shorter than originally planned. However, processing continued for one hour, allowing a 2.5 cm translation of the heater.

Post-flight analysis of the sample was completed. The following items were included in the reported conclusions:

(1) The space processing resulted in a single crystal 1 cm in diameter and approximately 7 cm long. (This length was much longer than the terrestrial theoretical length limit of a similar In-Tl float zone (0.74 cm).)

<Note: The following result was reported in Reference (2) and then appeared to be contradicted in Reference (3). Reference (2) stated:>

(2) "...the seed material was not a single crystal [as originally thought] and the orientation(s) of the seed sample is (are) unknown. Therefore, no conclusion can be drawn as to whether the single crystal formed during the experiment grew with the same orientation as the seed." (2, p. 144 (1987))

<Note: Information in Reference (3) somewhat contradicted item (2) (above) stating "...the portion of polycrystalline In that was melted and resolidified grew as a single crystal with the same orientation as the seed crystal." (3, p. 160 (1987)) Co-Investigator, M. D. Lind, verified that the information as stated in item 2 (Reference (2)) was correct (this specific Reference (3) information being incorrect).>

(3) The distribution coefficient of thallium in indium was found to be very small: the ratio of concentration in the solid to the ratio of concentration in the liquid was 0.03. Thus, "...the Tl was virtually removed from the melted and resolidified portion of the sample." (3, p. 160)

(4) Several unintentional impurities, thought to have originated from the glass tubes and other sources, were detected in the sample.

(5) An oxide layer was detected on both the ground-based samples and space-based sample. "It was concluded that an oxide layer sufficient to support the melted zone may have existed." (2, p. 145)

A discussion detailing possible Marangoni convection in the fluid portion of the sample was presented. It was noted that while oscillatory, surface tension-driven flows can be detrimental, laminar flows within the zone may produce a homogeneous distribution of impurities and dopants. Reportedly, "The uniformly small Tl concentration in the crystal grown during the STS-41D experiment shows that stirring was efficient." (2, p. 145) Because of the low Marangoni number within this experiment, the small surface tension gradient, and a small supposed boundary layer thickness for the Marangoni flow, it was expected that only steady Marangoni flows near the surface were created. Because indium oxide layers were found on the surfaces of both ground and flight

samples, it was thought that this oxide layer may have reduced Marangoni convection in the melt.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Resistance Heating, Float Zones, Binary Systems, Single Crystals, Seed Crystals, Feed Material, Dopant, Solutal Gradients, Concentration Distribution, Thermal Gradient, Boundary Layer, Free Surface, Surface Tension, Marangoni Convection, Marangoni Convection Diminished, Oscillatory Marangoni Convection, Thermocapillary Convection, Translation Rate, Solid/Liquid Interface, Crystalline Structure, Liquid/Gas Interface, Encapsulated Float Zones, Coated Float Zones, Contamination Source, Impurities, Thin Films, Oxide Layer, Hardware Malfunction, Incomplete Sample Processing

Number of Samples: one
Sample Materials: Polycrystalline indium (used as feed stock) doped with approximately 100 ppm thallium was joined to a seed crystal of indium.
(In*Tl*)
Container Materials: Hermetically sealed Pyrex^{TM} glass tube (2 cm diameter) filled with argon at a pressure of 1 atmosphere.

Experiment/Material Applications:
It was expected that space processing would allow much longer zone lengths than terrestrial processing. In turn, the larger zone "...allows more flexibility in heater design and control of the shape of the solid-liquid interface. It removes the requirement to rotate the ingot as in rf [sic] heating on Earth." (2, p. 139)

"Indium was chosen for study because its low melting point and the low vapor pressure of the melt at that temperature would simplify the apparatus and reduce the electrical energy and cooling requirements." (2, p. 139, abstract) It was also speculated that this liquid indium system would not experience thermocapillary convective flows.

References/Applicable Publications:
(1) Space Shuttle Mission 41-D, NASA Press Kit, June 1984, p. 24. (preflight)

(2) Murphy, S. P., Hendrick, M. J., Grant, R. W., and Lind, M. D.: Floating Zone Processing of Indium in Earth Orbit. Mat. Res. Soc. Symp. Proc., Vol. 87, 1987, pp. 139-147. (post-flight)

(3) Lind, M. D., Hendrick, J. J., and Martin, M. J.: Floating Zone Processing in Earth Orbit. Low Gravity Sciences, Vol. 67, Science and Technology Series, Edited by Koster, J. N., 1987. Published for the American Astronautical Society by Univelt, Inc., AAS Paper #86-557, pp. 149-163. (related research and post-flight results)

(4) Martin, M. J.: The Fluids Experiment Apparatus (FEA) and Rockwell's Industrial Space Processing Research Program. Prepared for presentation at the Materials Research Society 1986 Fall Meeting, Boston, Massachusetts, December 1, 1986, STS 86-0361, 8 pp. (experiment apparatus; post-flight)

(5) Fluids Experiment Apparatus FEA. Rockwell International Leaflet, Pub. #3544-U REV 3-88, 1 p. (experiment apparatus; post-flight)

(6) Fluids Experiment Apparatus. In Microgravity Science and Applications Experiment Apparatus and Facilities, document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 26-27. (processing facility)

(7) Hill, M. E. and O'Malley, T. F.: A Summary of Existing and Planned Experiment Hardware for Low-Gravity Fluids Research. AIAA 29th Aerospace Sciences Meeting, Reno, Nevada, January 7-10, 1991, p. 14, also NASA TM-103706. (post-flight)

(8) Personal communication with M. D. Lind, August 1993.

(9) The Purification and Growth of a Single Indium Crystal by the Float Zone Technique. In Shuttle Student Involvement Program (SSIP) Final Reports of Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-3: monoellipsoidal reflector furnace
Builder of Processing Facility: ERNO Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
Growth of a SiO_2 Coated Silicon Crystal from Melt Zone

When a silicon sample is rotated during float-zone crystal growth, rotational striations are found in the solidified, peripheral regions. These striations, generally millimeters apart, are produced when the crystal is rotated in an asymmetric temperature field. Between these rotational striations, non-rotational or microstriations are present with spacings on the order of microns. (Such non-rotational striations are also found in similarly produced materials which were not rotated.) These microstriations are caused by dopant inhomogeneities which result from fluctuations in the growth rate. The growth-rate fluctuations are caused by temperature fluctuations which occur due to oscillatory or turbulent flows in the melt. These time-dependent flows are driven by: (1) gravity-induced, buoyancy-driven convection (if density differences exist in the melt) and (2) gravity-independent, Marangoni convection (if surface-tension gradients exist in the melt).

On Earth, the individual contribution of these flows to striation formation is difficult to evaluate. However, under low-gravity conditions, buoyancy-driven flow is reduced and Marangoni flow dominates. Earlier, low-gravity studies (see Eyer's experiment on TEXUS 7 and Nitsche's and Eyer's experiment on Spacelab 1 (both in this chapter)) indicated that striations present in space-grown silicon crystals were caused by time-dependent, surface-tension driven Marangoni flows in the melt. To confirm these results, the effects of Marangoni convection were removed during this TEXUS 12 experiment by coating the float zone with a silica layer.

This TEXUS 12 experiment was the third in a series of investigations designed by Cröll and/or Nitsche and/or Eyer et al. to study the flow mechanism responsible for striations observed in float-zone solidified silicon (see Eyer, TEXUS 7, Nitsche, Spacelab 1). The specific objective of the experiment was to determine if purely diffusive conditions prevailed during the reduced-gravity float-zone growth of silica-coated silicon, or if laminar (non-striation creating) convection was present.

Prior to the TEXUS flight, a single phosphorous-doped Si crystal (90 mm long, 7.6 mm diameter) was boron-doped by filling a peripheral hole (1.5 mm depth, 1.2 mm diameter) with 0.45 mg of boron. After filling, the hole was closed by spot welding with focused light. (The boron dopant, released during melting, maps the diffusive or convective behavior of the melt.) The sample was then coated with a 5-micron-thick silica film by exposing it to a flow of oxygen saturated with water (50 hours at 1400 K). The sample was mounted free from the wall in a fused silica ampoule and sealed under 1.5 bar of oxygen to suppress hole formation in the silica film.

During the sounding rocket mission, the TEXUS monoellipsoidal reflector furnace was used to process the crystal using the float-zone method. A camera was included with the apparatus to allow real-time observation and control of the experiment. Prior to lift-off (time t = 0), the sample was preheated for 240 seconds. During lift-off the furnace was switched off to prevent damage to the lamp filament. At t = 65 seconds, the lamp power was increased to 600 W to assure sample melting. At t = 114 seconds, the power was reduced linearly to 450 W (this wattage had been used in ground-based experiments, resulting in a 17 mm zone length). Sample translation (5 mm/min) began at t = 160 seconds with the melt zone reaching the boron at t = 185 seconds (zone length = 18.5 mm). When the zone length reached 25.5 mm, the lamp was powered down in three steps to 410 W. (Apparently the lack of gravity-induced convection (which cooled the lamp and sample on Earth) resulted in the lower power requirement.) At t = 410 seconds, the power was switched off (total growth time = 250 seconds), resulting in a total recrystallized length of 35 mm (including a rapidly solidified zone of 23 mm).

<Note: Reportedly, a small portion of the crystal was grown while the sample rotated at a rate of 10 rpm for 1 minute. Figure 2 of Reference (3) indicates this began at about t = 65 seconds. However, this figure also implies that the rotation lasted much longer than 1 minute. No explanation or clarification of this discrepancy could be located. It was reported that the effect of this rotation was the occurrence of exactly ten rotational striations in the sample.>

A Si reference sample, prepared in the same manner as the flight sample, was processed on Earth. The terrestrial experiment parameters were identical to the flight sample up to t = 285 seconds. From this point to t = 410 seconds, a constant power of 450 W was used for ground-based processing.

Post-flight examination of the coated TEXUS Si crystal confirmed that microstriations found in earlier, low-gravity uncoated crystals were due to Marangoni convection effects. Analysis of the low-gravity processed crystal indicated that (1) the boron-doped area was limited in size and was separated by two adjacent n-type regions, and (2) the resistivity distribution in the flight sample was in excellent agreement with theoretical calculations which were determined assuming a purely diffusive dopant propagation.

In contrast, examination of the Earth-grown sample revealed a much larger boron doped region (than observed in the TEXUS sample). This result indicated that additional flow mechanisms had augmented the diffusive propagation. There was also a large deviation from the expected theoretical boron distribution. "Since Marangoni flow was excluded and since time dependent buoyancy flows should have produced striations, it is highly probable that the broadened boron distribution is caused by steady buoyancy flows." (4, p. 70)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Coated Float Zones, Encapsulated Float Zones, Binary Systems, Dopant, Single Crystals, Electronic Materials, Sample Rotation, Rotating Fluids, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Free Surface, Surface Tension, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Marangoni Convection Diminished, Time Dependent Thermocapillary Flow, Turbulent Flow, Buoyancy Effects Diminished, Liquid/Liquid Interface, Growth Rate, Solid/Liquid Interface, Liquid/Gas Interface, Concentration Distribution, Translation Rate, Striations, Rotational Striations, Sample Homogeneity, Coated Surfaces, Thin Films, Lamps

Number of Samples: one
Sample Materials: silicon doped with boron (Si*B*)

Container Materials: silica sample coating; fused silica crucible; SiO_2
(Si*O*)

Experiment/Material Applications:
Doped silicon is an important material used for the fabrication of integrated circuits. Variations in dopant levels, exhibited as striations, will cause localized variations in electronic properties. These variations can become problematic as the density of circuit devices on the integrated circuit increases. Understanding the formation of these striations will lead to more effective ground-based production of doped silicon crystals.
See also Eyer, TEXUS 7 (this chapter).

References/Applicable Publications:
(1) Cröll, A., Müller, W., and Nitsche, R.: Growth of an SiO^2 Coated Silicon Crystal. TEXUS 11/12 Abschlussbericht 1985, German publication, 1985. (in German)

(2) Input received from Experiment Investigator, May 1988.

(3) Cröll, A., Müller, W., and Nitsche, R.: Dopant Distribution in Semiconductor Crystals Under Microgravity. In Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 87-94.

(4) Cröll, A., Müller, W., and Nitsche, R.: Floating Zone Growth of Surface-Coated Silicon Under Microgravity. Journal of Crystal Growth, 79 (1986), pp. 65-70.

(5) Growth of a Si Crystal Covered with a SiO_2 Skin. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 176-177. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Mirror Heating Facility (MHF) Furnace: radiation from two halogen lamps is focused on sample by two ellipsoidal mirrors
Builder of Processing Facility: Dornier-System GmbH, Friedrichschafen, Germany

Experiment:
Floating Zone Crystallization of Silicon - A Comparison of the Striation Patterns of Space and Earth Crystals (WL-MHF-01)

This Spacelab D1 experiment was the fourth in a series of investigations designed by Cröll and/or Nitsche and/or Eyer et al. to study the flow mechanism responsible for striations observed in float-zone solidified silicon (see Eyer, TEXUS 7, Nitsche, Spacelab 1, Cröll, TEXUS 12 (all in this chapter)).

Two experimental runs were to be performed during the mission. The objective of the first run (designated as MHF 01A) was to confirm the results of an earlier Spacelab 1 experiment: that Marangoni flows may have caused striations in an uncoated crystal grown by the float-zone method (see Nitsche, Spacelab 1).

Reportedly, the "MHF 01A run" was canceled due to timeline problems." (2, p. 261)

The objective of the second run (designated as MHF 01B) was to investigate the differences in flow patterns found in coated Earth-processed and coated space-processed molten Si zones. This objective was to be accomplished by analyzing the distribution of a dopant released from a point source in the sample.

During the Spacelab D1 Mission, the Mirror Heating Facility (MHF) Furnace was used to process a phosphorous-doped Si rod (130 mm in length, 10 mm diameter). The intent of MHF 01B was to move a molten zone 15 mm long through the crystal at a rate of 5 mm/min. The center portion of the rod (approximately 70 mm in length) had been coated (on Earth) with a 5-micron-thick, coherent, amorphous

silica film. (Other portions of the rod had not been coated.) Within the coated section were two peripheral boron deposits (see Cröll, TEXUS 12 or Reference (2) for details of the sample coating procedures and boron-deposit procedures).

Reportedly, problems arose during the experiment run when an incorrect furnace power setting resulted in overheating of the sample. This power setting caused "...the zone length to increase unduly (approx. 24 mm instead of 15 mm). Video and data transmission for real-time correction was not available at this time. The excess zone length caused premature release of the boron deposits. Furthermore, the prolonged contact time between melt and coating and the higher temperature produced holes in the silica coating by the reaction: $Si + SiO_2 \rightarrow SiO$. Thus, the anticipated, diffusive conditions no longer prevailed because of Marangoni flows." (2, p. 261) <Note: It is not clear to the editors whether this reaction occurred over the entire length of the coated section or over a certain portion of the coated section. Examination of the composite surface topograms (Reference (2), p. 262 or Reference (4), p. 92) indicates striations in only a certain portion of the low-gravity sample.>

To obtain 1-g processed samples for comparison, two experiments were performed using the same time-power conditions as the flight experiment.

Post-flight analysis of the 1-g and low-gravity processed samples included (1) axial sectioning, (2) polishing and etching (modified Sirtl etch), (3) microphotography (Nomarsky interference contrast), and (4) resistivity measurements (1 mm intervals along rod axis with four-point probe). Comparison between surface topograms of the flight and 1-g reference samples led to the following conclusions:

(1) In both the 1-g and space crystals, the uncoated sample portions contained micron size striation patterns. In the coated portion of the space sample, no striations were present. This result confirmed the findings from previous low-gravity experiments that time-dependent Marangoni flows were responsible for striations in Si crystals. <Note: The following is not clear: if holes were present in the silica coating and thus Marangoni flows were present (as reported three paragraphs above), why were no striations found in the coated portions? It is suspected that there were still areas of the coated section which did not have holes.>

(2) The striation contrast in the uncoated 1-g processed sample was stronger than the contrast in the uncoated low-gravity processed sample. (This was attributed to additional buoyancy flow in the ground-based sample. The flow tended to decrease the

thickness of the diffusion layer at the interface in the ground sample, resulting in a greater effect of temperature fluctuations on the effective segregation coefficient.)

(3) Periodic striations (0.2-0.5 mm distances) were located in the 20 mm long coated portion of the 1-g processed sample. The striations were attributed to the transition from steady to oscillatory buoyancy flow.

Because of the overheating of the sample (excessive zone length), the desired, purely diffusive conditions could not be achieved. Resistivity versus axial distance curves indicated similar shapes for the 1-g and low-gravity processed samples. The calculated curve for the complete mixing case was also similar in shape to the those from the experimental samples.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Coated Float Zones, Encapsulated Float Zones, Binary Systems, Amorphous Materials, Dopant, Electronic Materials, Diffusion, Diffusive Mass Transfer, Free Surface, Free Surface Solidification, Free Surface Elimination, Surface Tension, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Time Dependent Thermocapillary Flow, Buoyancy Effects Diminished, Liquid/Liquid Interface, Growth Rate, Solid/Liquid Interface, Liquid/Gas Interface, Segregation, Segregation Coefficient, Concentration Distribution, Striations, Surface Morphology, Sample Homogeneity, Coated Surfaces, Thin Films, Liquid Film Rupture, Halogen Lamps, Sample Not Processed as Planned, Hardware Malfunction

Number of Samples: one
Sample Materials: Phosphorous-doped silicon with two point deposits of boron. The sample was partially coated with silica, SiO_2 (Si*P*B*, Si*O*)
Container Materials: unknown

Experiment/Material Applications:
See Cröll, TEXUS 12 (this chapter).

References/Applicable Publications:

(1) Cröll, A., Müller, W., and Nitsche, R.: Floating Zone (FZ) Crystallization of Silicon. In Scientific Goals of the German Spacelab Mission D1, WPF, pp. 109-111. (preflight)

(2) Cröll, A., Müller, W., and Nitsche, R.: Floating Zone Crystallization of Silicon. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 260-264. (post-flight)

(3) Input received from Experiment Investigator, May 1988.

(4) Cröll. A., Müller, W., and Nitsche, R.: Dopant Distribution in Semiconductor Crystals Under Microgravity. In Proc. 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 87-94.

(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements)

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Experiment Origin: France
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: The Gradient Heating Facility (GHF) Furnace used on Spacelab 1 was reused for this flight (with no hardware upgrades).
Builder of Processing Facility: CNES (Centre National d'Etudes Spatiales), Toulouse, France

Experiment:
Cellular Morphology in Lead Thallium Alloys (WL-GHF-02)

During directional solidification of a binary alloy, natural convection in the melt can affect the interfacial pattern of the alloy and thus affect the properties of the grown material.

This STS Spacelab D1 experiment was designed to investigate the effects of convection on cellular solidification. These effects were to be determined via a comparative study of cellular morphologies (1) on Earth (with a convecto-diffusive transport in the liquid phase) and (2) under low-gravity (with a diffusive-dominated transport in the liquid phase).

Lead-thallium alloys were chosen for the study because the threshold of morphological instability of this alloy system is weakly shifted by convection.

During the mission, three lead samples containing 25, 30 and 40 wt.% thallium were directionally solidified in the D1 Gradient Heating Facility (GHF). Post-flight, the samples were compared to three similar samples processed on Earth. A thermal gradient of 20 ^{o}C/cm and a growth velocity of 0.7 cm/h were chosen for all samples.

Reportedly, the space samples were affected by unexpected temperature fluctuations which occurred during the growth phase of the alloys. "It stems from these variations that, contrary to the 1g reference experiments, a stationary regime... [was never] achieved during the space experiment." (4, p. 232) Thus, the

solidification velocity was not constant. It was reported, however, that three-dimensional cells, which were regular and isotropic, were obtained. "Space samples exhibit[ed] homogeneous patterns, cells or dendrites depending on the value of the growth velocity...." (4, p. 233) In contrast, transverse sections of the upwardly-grown terrestrial samples exhibited complex structures (long striations plus cells) which were attributed to thermosolutal convection in the liquid.

The detailed comparison of space experiments with related cellular growth theories was limited by the unexpected temperature fluctuations during the low-gravity experiment. (The evolution of the solidification pattern in space is directly connected to the temperature profile.) Because the growth was not stationary in space, "...it is not possible to associate the instantaneous velocity to the spacing which was observed at the same time. The decrease in periodicity, which is concomitant with the decrease of the solidification rate indeed occurs very slowly and by a heavy process based on a massive division of the cells. This division begins by a tip splitting mechanism... which surely prohibits an immediate adaptation of the spacing." (4, pp. 234-235)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Cellular Morphology, Alloys, Binary Systems, Thermal Gradient, Solutal Gradients, Thermosolutal Convection, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Growth Rate, Solid/Liquid Interface, Interface Phenomena, Interface Physics, Concentration Distribution, Sample Microstructure, Striations, Dendritic Structure, Processing Difficulties

Number of Samples: three
Sample Materials: Lead-thallium samples containing 25, 30 and 40 wt.% thallium
(Pb*Tl*)
Container Materials: boron nitride
(B*N*)

Experiment/Material Applications:
Controlled solidification, which allows close regulation of the sample microstructure, and thus, the properties of the material, is a prominent technique employed in the production of engineering components. Therefore, it is important to determine the precise correlation between the microstructure and the processing conditions. Ground-based experiments have indicated that natural convection in the fluid system during the processing plays a significant role in the resultant sample microstructure.

The Principal Investigator reported that the objective of this low-gravity experiment was to provide reliable data for the solidification of massive specimens (to be used for testing the theories of pattern selection) under pure diffusion conditions. Unfortunately, the solidification velocity was actually not constant in space, which prevented these tests from being achieved.

The advantages of selecting the lead-thallium system are stated in the experiment summary.

References/Applicable Publications:
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(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements)

(6) Billia, B., Jamgotchian, H., Favier, J. J., and Camel, D.: Solidification cellulaire d'alliages Pb-Tl lors de l'expèrience D1-WL-GHF-02. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity, ESA, Noordwijk, 1987, pp. 377-381. (in French)

(7) Input received from Principal Investigator B. Billia, July 1989 and July 1993.

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Experiment Origin: USA
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Experiment Assembly (MEA-A2) located in the STS payload bay
Processing Facility: Gradient General Purpose Rocket Furnace (G-GPRF)
Builder of Processing Facility: NASA Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Semiconductor Materials-Suppression of Body Force Gravity-Driven Convection in Directionally Solidified PbTe-SnTe (ME-GPRF 5)

During ground-based, directional solidification, gravity-induced convection in semiconductor melts contributes to crystalline defects such as low angle grain boundaries and dislocations. These defects, in turn, severely degrade the overall performance of instruments which employ the crystals.

The objective of this STS directional solidification experiment was to determine to what extent convective mixing would be reduced during the space processing of a lead-tin telluride alloy.

During the mission, the semiconductor material (composed of 20% SnTe and 80% PbTe) was processed in the Gradient General Purpose Rocket Furnace (G-GPRF). The furnace had a maximum operating temperature of approximately 1000 ^{o}C which accommodated the processing temperature of the sample (904 ^{o}C). Directional solidification was achieved in the G-GPRF via (1) three independently programmable temperature zones and (2) a water-cooled heat exchanger located at one end of the furnace. No ampoule or furnace translation was required (or possible) using this G-GPRF experimental setup.

Reportedly, "Furnace characterization is important for any crystal growth experiment but it is especially vital for a stationary furnace such [as the] GPRF in which a temperature gradient is to

be translated through the furnace at a constant rate. To this end an instrumented SiO_2 cartridge, with thermal properties similar to lead tin telluride, was constructed and ground based tests were conducted in the flight furnace. The maximum temperature gradient near 900 ^{o}C that could be maintained over the length of the cartridge was 20^{o}C/cm, hence the growth rate needed to be below 3.5 mm/hr to prevent interfacial breakdown. The growth rate was derived by carefully profiling the furnace at various zone settings and then, after the cooling water was turned on to initiate growth, determining both the zone temperatures and the time between these settings which would translate a 900^{o}C isotherm through the cartridge at a constant 3.5 mm/hr." (7, p. 332) Diffusion-induced homogenization was to be realized before directional solidification was initiated.

Preliminary post-flight analysis of the outer surface of the processed space crystal indicated that it was "...slightly grainy, with a few small grains, on the order of a millimeter square contacting the ampoule, and the rest of the surface very near the wall. The sample was not stuck to the walls of the ampoule. There was an unusual feature on the last to freeze surface of the sample that appeared to be a small bump, about five millimeters in diameter. Lead tin telluride had a crystal structure that solidifies at a higher density than the liquid, i.e. the volume of the solid is less than the volume of the liquid. If the melt had wet the ampoule, it was possible therefore, that voids would be formed in the space grown crystal. Radiography of the sample before the ampoule was opened showed that no voids were present." (7, p. 333)

Preliminary analysis of the axial composition of the sample indicated that considerable convective mixing had occurred during the solidification of the sample material. Thus, planar, diffusion-controlled growth did not result as expected. It was speculated that this mixing within the melt was produced by residual acceleration forces (less than 10^{-4} g).

Analysis of the furnace operation indicated "...the cooling water loop did not function as planned.... The lack of coolant, of course, changes the growth rate. Additional calibration runs are being conducted to determine the actual growth rates that occurred under flight conditions. In addition, post flight analysis has shown that rather than being under vacuum conditions, the furnace was most likely filled with argon. This would also cause a significant change in the heat transfer characteristics." (7, p. 334)

Key Words: Crystal Growth From the Melt, Melt and Solidification, Bridgman Technique, Directional Solidification, Ternary Systems, Semiconductors, Semiconductor Applications, Electronic Materials, Infrared Detector Applications, Thermal Gradient, Solutal Gradients, Convection, Thermosolutal Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Diffusion, Diffusion-Controlled Growth, Diffusive Mass Transfer, Wetting, Non-Wetting of Container, Interface Physics, Interface Phenomena, Growth Rate, Solid/Liquid Interface, Planar Solidification Interface, Surface Morphology, Density Distribution, Sample Homogeneity, Crystalline Defects, Crystalline Structure, Grain Boundaries, Dislocations, Acceleration Effects, Volume Change, Sample Deformation, Sample Detachment From Crucible, Free Surface, Voids, Vacuum, Furnace Malfunction

Number of Samples: one
Sample Materials: lead-tin-telluride
(Pb*Sn*Te*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
"Lead tin telluride is a substitutional alloy of lead telluride and tin telluride that is miscible over the entire compositional range. It has a phase diagram similar to other compound semiconductors of interest, such as mercury cadmium telluride and mercury zinc telluride; however unlike these materials, it is thermo-solutally unstable in a 1-g environment... lead tin telluride is amenable to study since [1] it is easily compounded, [2] it has a relatively low vapor pressure, [3] it is single phase in the solid and [4] there is a sufficient amount of previous results on its growth and thermophysical properties to allow thermal modeling of the experiments." (7, p. 331)

This directional solidification research is applicable to the growth of crystals for use in infrared detectors and tunable diode lasers. Reportedly, such devices "...have not yet been optimized for peak performance." (4, p. 2)

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(3) Fripp, A. L. and Debnam, W. J.: Microgravity Crystal Growth on Mission 61A. In Research and Technology 1987, Annual Report of the Langley Research Center, p. 64, NASA TM-4021. (very short description)

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Experiment Origin: France
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, ESA Materials Science Double Rack (MSDR)
Processing Facility: Gradient Heating Facility (GHF)
Builder of Processing Facility: Centre d'Etudes Nucléaires (CENG)/Centre National d'Etudes Spatiales (CNES), Toulouse, France

Experiment:
Growth Rate Measurement of Doped InSb Crystal by Method Under Microgravity (WL-GHF 03)

Access to the low-gravity environment for solidification studies provides new possibilities for indepth control of the resulting solidified structure. This control requires in situ measurement of various solidification parameters. One method of monitoring the solidification process is based on the calorimetric principle. Theoretically, the growth rate of a solidification interface may be calculated from (1) the latent heat of solidification, (2) the thermal flux in the liquid, and (3) the thermal flux in the solid. Since it is not possible to measure the thermal fluxes on the interface itself, these determinations must be made at some distance from the interface, with the expected heat diffusion and heat loss accounted for. Under low-gravity conditions, the characteristic of diffusive heat transport (absence of convection) allows testing of the calorimetric principle.

This Spacelab D1 experiment was the third in a series of investigations designed by Potard et al. to study low-gravity directional solidification (see Potard, SPAR 9 (Chapter 17), Spacelab 1 (Chpater 4)). Specifically, this experiment was designed to test and validate a calorimetric method for determining the growth rate of (1) Sn-doped InSb crystals and (2) pure InSb crystals. The objectives of the study were to determine (1) the heat flux intensity, (2) the heat flux variation with solidification

parameters, and (3) the instantaneous heat content of the sample.

During the mission, three InSb samples (two pure samples and one heavily doped with tin) were directionally solidified in the Spacelab Gradient Heating Facility (GHF). Each sample was configured in a quartz crucible which also contained (1) the seed material and (2) a graphite block through which heat was injected. The heat flux in each sample was measured with (1) a thermocouple-pair located in the seed material and (2) a thermocouple-pair located in the graphite block.

In the low-gravity environment, both wetting and non-wetting of the crucible by the melt can occur. "These physical contact fluctuations associated with free liquid surface may be [a] source of thermal fluxes sharing, between the sample and crucible." (5, p. 269) To determine these fluctuations, the sample and crucible were subjected to certain well-known boundary conditions: (1) controlled temperature at the ends and (2) an adiabatic crucible surface. For calibration and capacity evaluation, the sample and crucible were subjected to certain stimuli: (1) a sharp area change of the crucible and sample and (2) for the Sn-doped sample, constitutional destabilization of the interface. (See Reference (5) for further experiment details.)

The results from this experiment were evaluated in conjunction with numerical simulations (see Reference (6)) and ground-based studies (see Reference (5)).

Post-flight analysis of the data and samples revealed that calorimetric measurements can be utilized for real-time monitoring of the crystallization process. However, it was reported that "The sample ends temperatures [were] obtained for two separate samples without the corresponding flux measurement... and the fluxmeter data for the doped sample only. Fortunately, the parallel processing method applied to the three samples permitted [development of] a near-normal exploitation step." (5, p. 273) Despite these difficulties, the seeding, transient, and steady state directional solidification conditions were identified utilizing this calorimetric method. However, it was indicated that practical application of this procedure requires (1) avoidance of radial thermal fluxes between fluxmeters, (2) flux measurements at locations of flat isotherms, and (3) numerical simulation for accuracy determination.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Steady-State Solidification, Dopant, Ternary Systems, Binary Systems, Sample Purity, Seed Crystals, Calorimetric Measurements, Thermal Gradient, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Heat Flux, Buoyancy Effects Diminished, Growth Rate, Solid/Liquid Interface, Interface Physics, Interface Stability, Solidification Front Physics, Wetting, Wetting of Container, Crucible Effects, Container Shape, Hardware Malfunction

Number of Samples: three
Sample Materials: pure indium-antimony and tin-doped indium antimonide
(In*Sb*, In*Sb*Sn*)
Container Materials: silica
(Si*O*)

Experiment/Material Applications:
To achieve a high degree of reliability in space processing techniques, in situ measurement and control of solidification parameters are necessary. The calorimetric method was evaluated to determine its feasibility for real-time measurement of controlled solidification parameters.

The specific reason why the InSb alloys were selected for this experiment was not detailed in available publications.

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(1) Potard, C. and Duffar, T.: Doped In-Sb Crystallization. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, August 27-29, 1986, p. 87. (postflight)

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(6) Duffar, T., Potard, C., and Dusserre, P.: Growth Analysis of the InSb Compound by a Calorimetric Method in Microgravity; Results of the Spacelab D1 Experiment. Journal of Crystal Growth, Vol. 92, 1988, pp. 467-478. (post-flight)

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Co-Investigator(s): None
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Experiment Origin: Canada
Mission: STS Launch #23, STS-031 (STS 61-B, Atlantis)
Launch Date/Expt. Date: November 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-479
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of this experiment within G-479: National Research Council of Canada (NRC), Ottawa, Ontario, Canada/Telesat Canada, Ontario, Canada
Processing Facility: Glass containers housing cadmium rods which were heated. Insulation of the containers insured the correct processing temperature gradient.
Builder of Processing Facility: A team of scientists and engineers at the National Research Council of Canada, Ottawa, Ontario, Canada

Experiment:
Cadmium Crystal Production

This experiment was one of two investigations housed within the G-479 Get Away Special canister during STS-031. The other experiment of the two is also applicable to this database (see Rey, STS-031 "Primary Mirror Production Using Vapor Deposition" (Chapter 10)).

This crystal production experiment, which was designed, built, and tested by scientists and engineers at the National Research Council of Canada, was integrated into the G-479 GAS canister with the Telesat Canada Primary Mirror Production Experiment when it was determined that the mirror experiment configuration (1) left unused space in the canister and (2) had excess battery capacity to power a second experiment.

The experiment proposal was accepted by Telesat Canada in June 1984 (the canister was scheduled to fly just 4 months later on the shuttle in October 1984). Several constraints (dictated by the primary mirror production experiment already within the GAS can) had to be addressed prior to experiment assembly: volume, weight, operating temperature, power (65 W), etc. Nevertheless, 2 months later, the completed experiment assembly was integrated into the G-479 canister ready for the October launch. Eventually, the canister was removed from the originally scheduled flight in order to clear the cargo bay for the rescue of two

satellites. The canister was finally flown (no upgrades allowed) in November 1985.

The experimental concept was based on (1) the Principal Investigator's scientific experience with cadmium crystals, (2) Walter's earlier Skylab InSb quasi-containerless crystal growth experiments (see Walter, SL-3 and SL-4 (Chapter 6)) and (3) Witt's ASTP Ga-doped Ge experiment (see Witt, ASTP (this chapter)).

During the earlier Skylab experiment, a containerless section of an InSb cylindrical sample was melted in a gradient furnace and resolidified. Although it was expected that the solidified free section of the crystal would be spherical, the crystals grew into a longer tear-drop shape (containing facets around their circumference) and unexpectedly made contact with the container wall. During the earlier ASTP experiment, "...it was found that gallium-doped germanium crystals regrown in a closely fitting container (in the bridgman method) did not contact the container as they would have on earth, but developed 'ribs' which contacted only about 1% of the container surface. Thus a form of containerless growth was achieved without special measures." (8, p. 55)

Based on the three factors above, the specific objective of the experiment was to determine if polycrystalline cadmium rods would behave similarly to the crystals flown during these earlier experiments.

Prior to the shuttle flight 10 cadmium specimens were prepared. "The metal was purified, mainly for the removal of oxides, by the casting in a vacuum. The castings were used as billets for the hot extrusion of 2 mm diameter rods in a press. The rods, cut to length became the specimens [each specimen 44 mm long]. No special precautions were taken to clean the surface of the rods before sealing into a container [with an argon atmosphere], an omission which had notable consequences."

"The containers were designed so that the cadmium rod would not be supported everywhere, but would be held at one end and either stand free for the remainder of its length or be in some form of contact with the inside of the container. The latter portion of the container would be heated until the metal melted and then cooled to permit resolidification." (8, pp. 55-56)

The specimen containers were made from two standard sizes of Pyrex™ tubing - 6 mm o.d. and 10 mm o.d. - blown to the the shape desired. Some of the containers had circular cross-sections (such that no section of the sample's free section would touch the container). The other containers had circular cross-

sections with either three or four equally spaced indentations made during container fabrication by pressing the softened Pyrex™ inwards with a chuck along the length of the tubing in three or four equally spaced positions around the tube circumference (such that the sample would touch the tube at three or four longitudinal locations, see Reference (8), Figure 1.) The specimens were anchored at one end by melting the end and shrinking the tubing over the form.

Bundles of containers "...were assembled by wrapping them with Nicrome ribbon, which served both to secure and heat them. Asbestos tape was wrapped around the outside and secured with cement. Finally aluminum foil was wrapped around the outside and secured with wire. Since only free-standing [or semi-free standing] section of the specimen was melted, resolidification could start from the unmelted part." (8, p. 56)

Terrestrial experiments were performed to determine the power required to melt the cadmium bundles. "If too little power was used the cadmium might not melt, while if too much was used the cadmium might melt back into the bulb. It was therefore decided to cut off the power when a certain temperature, indicated by the thermocouple voltage was reached. The temperature chosen was about 10 ^{o}C over the melting point." (8, p. 57)

During the shuttle mission, power was supplied to the experiment for 10 min, 40 seconds. Since time to fuse blowout on Earth was about 8 minutes, it was thought that the experiment might not have proceeded as planned "...since the survival of the fuse showed that the controlling thermocouple had not reached its cutoff temperature." (8, p. 57)

Post-flight examination of the flight specimens indicated that (1) all of the eight specimens in the 6 mm o.d. containers had melted to some degree (although not to the degree desired) and (2) the two specimens in the 8 mm o.d. containers had not melted. Reportedly, the samples did not melt as expected because of an inadequate estimate of convective heat transfer in space.

The general appearance of the flight specimens was much the same as before the flight. "Although unexpected[,] the near retention of the original shape proved due to the presence of an oxide coating, left from the [ground-based, preflight] extrusion process, which acted as a container in microgravity...." (8, p. 55) Soft X-ray wavelength dispersive analysis showed that the oxide layer was approximately 100-170... angstroms thick-substantially more than a normal layer on earth." (8, p. 55)

"The melted lengths, determined with a measuring microscope, ranged from 5 to 28.5 mm [the expected meltback was 25 mm]. There appears to be no firm correlation between the number of indentations and melted length although containers with indentations in general had longer melted lengths than those without. Grain size was in general larger with increased melted length, but the maximum diameter shows no relation. Only the solid-melt interface changed in a regular way with irregular interfaces for short melted lengths and precise circular melts for longer lengths." (8, p. 58) All melted specimens exhibited a slight increase in diameter.

"The grain structure of the specimens was revealed by etching. X-ray photographs were taken with Laue back reflection to show the crystal structure. The grains were large in some specimens, smaller in others, but consistently larger than grains in the unmelted metal." (8, p. 55)

It was concluded that:

"Although prepared in haste, the experiment showed several phenomena not to be seen on earth. The external shape of the specimens showed that the metal remained clear of the container walls and that an oxide layer remaining from extrusion of the rods acted as a slightly elastic container. The surface of the melted length did not retain die marks, although traces remained on some specimens. The grain size of the specimens varied greatly, but was always larger than that of the starting material. The crystallinity of the specimens ranged from highly perfect to greatly imperfect. Nucleation occurred both at grains at the solid-melt interface and at the tips of some specimens." (8, p. 61)

A detailed description of several of the samples is provided in Reference (8).

Key Words: Crystal Growth From the Melt, Metals, Melt and Solidification, Semi-Containerless Melt, Float Zones, Encapsulated Float Zones, Coated Float Zones, Free Surface, Surface Tension, Wetting, Non-Wetting of Container, Sample Detachment From Crucible, Buoyancy Effects Diminished, Absence of Buoyancy Forces (Detrimental), Heat Transfer, Solid/Liquid Interface, Liquid/Gas Interface, Interface Shapes, Oxidation, Oxide Layer, Coated Surfaces, Thin Films, Contamination Source, Sample Purity, Surface Morphology, Crystalline Structure, Nucleation, Grain Size, Volume Change, Volume Expansion, Thermal Environment More Extreme Than

Predicted, Sample Not Processed as Planned, Inomplete Sample Processing

Number of Samples: ten samples were to be melted
Sample Materials: cadmium rods
(Cd*)
Container Materials: glass (Pyrex^{TM})

Experiment/Material Applications:
See Walter, Skylab SL-3 (Chapter 6).

Cadmium was selected for this experiment because (1) the Principal Investigator was familiar with the material, (2) it has a low melting point (321 ^{o}C), (3) it can be contained in and does not wet Pyrex^{TM} (Pyrex^{TM} is easy to mold and work with), and (4) the crystal structure is hexagonal close packed (in contrast to the cubic structure of InSb and Ge).

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(5) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA GAS Team, 1989, p. 32. (post-flight; very brief description)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(7) Personal communication with Principal Investigator F. Lipsett, September 1993.

(8) Lipsett, F. and Sewell, P. B.: Resolidification of Cadmium in Space and the Role of an Oxide Coating as a Container. Appl. Microgravity Technology, 1, Vol. 2, 1988, pp. 55-61. (post-flight)

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Experiment Origin: Canada (sponsored by the Canadian Space Agency)
Mission: MASER 1
Launch Date/Expt. Date: March 1987
Launched From: Esrange, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: GEODE furnaces: two directional solidification furnaces, each configured with six individual resistor heaters. The heaters were controlled by a microprocessor.
Builder of Processing Facility: The Electrofuel Manufacturing Company, Toronto, Ontario, Canada

Experiment:
GEODE: Gravity Experiment on Detector Elements

On Earth, gravity-driven convection and material constituent segregation contribute to compositional inhomogeneity of single crystals. The objective of this MASER sounding rocket experiment was to improve the compositional homogeneity of CdHgTe by solidifying the material in a reduced gravity environment.

Prior to the rocket flight, two 95 gm samples of $Cd_{0.21}Hg_{0.79}Te$ were prepared. Each of the samples were sealed in an evacuated quartz ampoule. "The ampoule design consisted of two cylindrical sections with different diameters [15 mm diameter i.d. and 7.0 mm i.d.]. Since molten $Cd_{0.21}Hg_{0.79}Te$ does not wet quartz, the melt was expected to fill the near-zero free volume of the 15 mm diameter section due to surface tension." (2, p. 58) "The 15 mm inside diameter, 1.5 mm wall thickness section provided the maximum sized wafers considered possible because of ampoule strength limitations required to contain the high pressure. The 7 mm I.D. section enabled ampoule sealing and free volume reduction." (Reference (6))

The experiments were performed in two similar gradient (directional solidification) furnaces. Each furnace was configured with six individual resistor heaters to produce six heating zones. Reportedly, "Each furnace... [was] controlled by an onboard microprocessor which... [was] programmed to control the power applied to each temperature zone heater. The different zone temperatures... [were] maintained or altered according to

the required temperature profile." (1, p. 19)

Just prior to the rocket launch, the samples were heated to a molten state (10 ^{o}C above the liquidus temperature). <Note: It was stated that the homogenization temperature of the samples was 820 ^{o}C.>

Just after the start of the low-gravity phase of the mission, solidification of the sample was initiated. "Quenching was accomplished by directing N_2 gas at the tip of the ampoule through the annular cavity surrounding the ampoule, and then vented externally through an exhaust manifold." (2, p. 58)

Reportedly, "Twelve short pulses of up to 4 X 10^{-4}g were recorded by on-board accelerometers" (2, p. 58) during the low-gravity phase of the rocket flight.

Post-flight examination of the payload indicated that the furnaces operated essentially as expected. "However, in one of the furnaces, the quartz ampoule containing the sample broke, probably during launch. The other sample survived the flight and... [was expected to yield] good results." (1, p. 19)

The characteristics of the surviving sample were compared with similarly processed ground-based reference samples. "The samples were cut into five sections. The first, third and fifth sections were left in the as-quenched state. The second and fourth sections were recrystallized in a quartz ampoule having a Hg vapour compensated atmosphere.

"The single crystal volume in each of the recrystallized test sections [MASER and terrestrial] was approximately 50%. These sections were wafered...." (2, p. 58) (Additional information on post-flight sample procedures can be found in Reference (2).)

Infrared transmission data was collected using Analect Instruments' AQS-20 FT-IR spectrometer. CdTe-mole-fraction vs. relative-ingot-position graphs were plotted for the samples to illustrate axial composition profiles. Each of these graphs had three curves of the mole fraction value, x: (1) the maximum composition value (typically measured on the outside edge of the sample wafer), (2) the minimum concentration value at the center of the sample wafer, and (3) the target melt stoichiometry of x = 0.21 (straight line). The difference between curves (1) and (2) indicated the maximum segregation in the sample.

The terrestrial baseline sample illustrated a large difference between curves (1) and (2) (and thus a large degree of segregation) especially at the tip of the ingot (first to solidify). The degree of segregation decreased monotonically

along the growth axis. In contrast, the degree of segregation in the flight sample was significantly less. Reportedly, "The average composition throughout the analyzed regions of the crystal was 0.211 +/- 0.003." (2, p. 60)

CdTe-mole-fraction vs. relative-ingot-position graphs were plotted for the samples to illustrate radial composition profiles as well. (Please refer to Reference (2), Figure 5.) It was concluded that the IR data indicated that the flight sample had "...significant improvement in both radial and longitudinal uniformity..." (2, p. 57) when compared to the reference samples.

Comparison of the compositional structure for the terrestrial and flight samples (using wafer contour plots to show radial symmetry) can be seen in Reference (2), Figure 6. <Note: the text of Reference (2) indicates that the figure number is 6, but the figure itself is labeled 5a, 5b, 5c, and 5d.> It was reported that "There were no observed composition structure anomalies, as observed by transmission analysis, in the space grown sample that could be attributed to the twelve [previously mentioned] accelerometer peaks. There may be some evidence of these disturbances in the as-grown material, but the characterization of these sections has not been completed." (2, p. 60)

<Note: References (7) and (8) were not available at the time this experiment summary was prepared. They may contain additional information concerning the experiment.>

Key Words: Crystal Growth From the Melt, Melt and Solidification, Directional Solidification, Resistance Heating, Single Crystals, Semiconductors, Electronic Materials, Infrared Detector Applications, Infrared Transmission, Composition Distribution, Composition Variation, Crystal Homogeneity, Sample Homogeneity, Segregation, Axial Segregation, Buoyancy-Driven Convection, Thermal Gradient, Solid/Liquid Interface, Interface Physics, Solidification Front Physics, Wetting, Non-Wetting of Container, Surface Tension, Volume Change, Quench Process, Rocket Motion, Acceleration Effects, Acceleration Measurements, Payload Survivability, Vacuum

Number of Samples: two
Sample Materials: 95 gm samples of $Cd_{0.21}Hg_{0.79}Te$
(Cd*Hg*Te*)
Container Materials: quartz
(Si*O*)

Experiment/Material Applications:
"$Cd_xHg_{1-x}Te$ forms a compositionally variable material system with a tunable infrared wavelength sensitivity. The variable, x, refers to the CdTe mole fraction. Near theoretical detection efficiency has been demonstrated for the radiation range of 1-40 micrometers." (2, p. 57)

This low-gravity research was performed to reduce gravity-driven convection and segregation in the molten CdHgTe sample. If improved compositional homogeneity was demonstrated, improved infrared detectors could be possible.

References/Applicable Publications:
(1) Zaar, J. and Änggard, K.: MASER and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987 Japan Space Utilization Promotion Center (JSUP), 32 pp. (post-flight; short description)

(2) Bollong, A. B. and Proux, G. T.: Analysis of $Cd_{0.21}Hg_{0.79}Te$ Quenched in 10^{-4} g. In Proceedings Workshop on Microgravity Experimentation in Aircraft and Rockets, May 5-6, 1988, Ottawa, Canada, pp. 57-61. (post-flight)

(3) Jönsson, R.: The Microgravity Program in Sweden-Emphasis on the Materials Rocket Maser. In 15th International Symposium on Space Technology and Science, Tokyo, Japan, May 19-23, 1986, pp. 2099-2110. (preflight)

(4) Zaar, J., Björn, L. and Jönsson, R.: Preliminary MASER 1 Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short summary)

(5) Jönsson, R.: SSC Microgravity Sounding Rocket Program MASER. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(6) Input received from Experiment Investigator, December 1989 and August 1993.

(7) Redden, R. F., et al.: Infrared Detection-Mercury Cadmium Telluride and Microgravity. In Proc. Spacebound '93, May 16-18, 1993, Ottawa, Ontario. <Note: The page numbers of this article were not identified by the Experiment Investigator.>

(8) Bollong, A. B. and Proux, G. T.: Analysis of $Cd_{0.21}Hg_{0.79}Te$ Quenched in 10^{-4} g. Journal of Crystal Growth, 94, 1989, pp. 475-480.

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 15
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-3
Builder of Processing Facility: ERNO Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
GaAs Crystallization

This TEXUS 15 experiment was the first in a series of low-gravity experiments designed by G. Müller to examine the crystallization of gallium-arsenide.

<Note: Details of the research objectives and experimental setup of this TEXUS 15 experiment were not presented in the available publications. However, Principal Investigator, G. Müller, indicated that this experiment and his later TEXUS 16 experiment (see Müller, TEXUS 16) were similar to his experiments performed on the TEXUS rocket in 1988 (TEXUS 20) and 1989 (mission number not specified). (The TEXUS 20 and later mission results are summarized in Reference (3).) The following summary is based primarily on Reference (3) and the Principal Investigator's response.>

The objectives of the research were to (1) test the feasibility of processing large diameter GaAs crystals by the float-zone method under the low-gravity conditions, (2) numerically predict the thermal conditions which will lead to time-dependent convection in the crystals, and (3) analyze the microscopic dopant distribution of 1-g and low-g processed GaAs crystals to determine the effect of Marangoni convection in the melt (the occurrence of striation patterns can usually be related to time-dependent convection).

The size (diameter) of the GaAs rod employed for the experiment was limited by the short processing time available on TEXUS under low-gravity (6-7 minutes). A rod diameter of 10 mm was selected such that the entire crystal growth length would be at least a few millimeters long.

Prior to the mission, a single GaAs sample was configured in a monoellipsoid mirror furnace. During the mission, a stable floating zone (without a solid core) was to be established, followed by crystal pulling.

Reportedly, shortly after the successful launch of the TEXUS 15 rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burnthrough of the second stage of the rocket had occurred and the stage, in turn, had collided with the prematurely separated payload. The upper part of the payload, including the TEM 02-3 module, parachuted to the Earth undamaged.

<Note: The Principal Investigator implied (Reference (2)) that the results from this experiment confirmed that unsteady Marangoni convection was present in the melt and that this convection created doping striations. However, it is not clear if he was actually referring to the later 1988-1989 experiments.>

Documentation further detailing any results of this experiment does not appear to be available.

<Note: Müller's later TEXUS experiments (TEXUS 20 et al.) are beyond the scope of this Technical Memorandum (TM). These later experiments will be contained in later versions of this TM.>

Key Words: Crystal Growth From the Melt, Melt and Solidification, Free Surface Solidification, Single Crystals, Float Zones, Float Zone Stability, Dopant, Binary Systems, Semiconductors, Electronic Materials, Optics Applications, Sample Purity, Heat and Mass Transfer, Free Surface, Surface Tension, Thermal Gradient, Time Dependent Thermocapillary Flow, Thermocapillary Convection, Marangoni Convection, Buoyancy Effects Diminished, Solid/Liquid Interface, Liquid/Gas Interface, Striations, Crystal Homogeneity, Crystalline Defects, Containerless Processing, Rocket Motion, Acceleration Effects, Rocket Failure, Payload Survivability

Number of Samples: one
Sample Materials: gallium-arsenide
(Ga*As*)
Container Materials: silica ampoule (no contact with melt)
(Si*O*)

Experiment/Material Applications:
"GaAs is the most important semiconducting material for the production of optoelectronic devices and gains increasing interest for high speed electronic devices. But the further development in this... [field] of application is impeded by quality problems of the GaAs single crystals. The most important of these problems are: crystal defects, impurities and microinhomogeneities.... Crystal defects could be reduced and the purity can be improved... [when] there is no interaction between the melt and the growing crystal with the crucible....

"But because of the limitations set by the hydrostatic pressure... crystals with diameter > 8 mm can be grown by this technique under microgravity conditions. Furthermore, buoyancy driven convection which is the most important cause of microinhomogeneities (striations) is absent under microgravity conditions. But unfortunately the presence of free melt surfaces can cause another type of convection, the so-called Marangoni convection (MC). This type of convection is driven by gradients of the surface tension (due to temperature or chemical gradients). The MC can become time-dependent if the driving forces exceed a certain value similar to buoyancy driven convection. Presently very few experimental results exist concerning the transition from steady to unsteady MC in liquid metals and semiconductor melts...." (3, p. (7)297).

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 107-108. (in German; post-flight)

(2) Input received from Principal Investigator G. Müller, August 1993.

(3) Rupp, R., Auerochs, A., Müller, G., Weyrich, C., and Leibenzeder, S.: Growth of GaAs Single Crystals by the Floating Zone Technique Under Microgravity. Adv. Space Res., Vol. 11, No. 7, 1991, pp. (7)297-(7)304. (discusses later TEXUS efforts; does not specifically mention this or the TEXUS 16 experiment)

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 02-3
Builder of Processing Facility: ERNO Raumfahrttechnik GmbH, Bremen, Germany

Experiment:
GaAs Crystallization

This TEXUS 16 experiment was the second in a series of low-gravity experiments designed by G. Müller to examine the crystallization of gallium arsenide (see Müller, TEXUS 15).

<Note: Details of the research objectives and experimental setup of this TEXUS 16 experiment were not presented in the available publications. However, Principal Investigator, G. Müller, indicated that this experiment and his earlier TEXUS 15 experiment (see Müller, TEXUS 15) were similar to his experiments performed on the TEXUS rocket in 1988 (TEXUS 20) and 1989 (mission number not specified). (The TEXUS 20 and later mission results are summarized in Reference (3).) The following summary is based primarily on Reference (3) and the Principal Investigator's response.>

The objectives of the research were to (1) test the feasibility of processing large diameter GaAs crystals by the float-zone method under the low-gravity conditions, (2) numerically predict the thermal conditions which will lead to time-dependent convection in the crystals, and (3) analyze the microscopic dopant distribution of 1-g and low-g processed GaAs crystals to determine the effect of Marangoni convection in the melt (the occurrence of striation patterns can usually be related to time-dependent convection).

The size (diameter) of the GaAs rod employed for the experiment was limited by the short processing time available on TEXUS under low-gravity (6-7 minutes). A rod diameter of 10 mm was selected such that the entire crystal growth length would be at least a few millimeters long.

Prior to the mission, a single GaAs sample was configured in a monoellipsoid mirror furnace. During the mission, a stable floating zone (without a solid core) was to be established, followed by crystal pulling. In contrast to the TEXUS 15 experiment, an As-vapor pressure source was employed.

Reportedly (Reference (1)), shortly after the successful launch of the TEXUS 16 rocket, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including the TEM 02-3 module.

Documentation further detailing any results of this experiment does not appear to be available.

Key Words: Crystal Growth From the Melt, Melt and Solidification, Single Crystals, Float Zones, Float Zone Stability, Dopant, Binary Systems, Semiconductors, Electronic Materials, Optics Applications, Heat and Mass Transfer, Free Surface, Surface Tension, Thermal Gradient, Time Dependent Thermocapillary Flow, Thermocapillary Convection, Marangoni Convection, Buoyancy Effects Diminished, Solid/Liquid Interface, Free Surface Solidification, Liquid/Gas Interface, Striations, Crystal Homogeneity, Containerless Processing, Rocket Motion, Acceleration Effects, Rocket Failure, Payload Recovery System Failure, Payload Survivability

Number of Samples: one
Sample Materials: gallium-arsenide
(Ga*As*)
Container Materials: silica ampoule (no contact with melt)
(Si*O*)

Experiment/Material Applications:
"GaAs is the most important semiconducting material for the production of optoelectronic devices and gains increasing interest for high speed electronic devices. But the further develop-

ment in this... [field] of application is impeded by quality problems of the GaAs single crystals. The most important of these problems are: crystal defects, impurities and micro-inhomogeneities.... Crystal defects could be reduced and the purity can be improved... [when] there is no interaction between the melt and the growing crystal with the crucible....

"But because of the limitations set by the hydrostatic pressure... crystals with diameter > 8 mm can be grown by this technique under microgravity conditions. Furthermore, buoyancy driven convection which is the most important cause of micro-inhomogeneities (striations) is absent under microgravity conditions. But unfortunately the presence of free melt surfaces can cause another type of convection, the so-called Marangoni convection (MC). This type of convection is driven by gradients of the surface tension (due to temperature or chemical gradients). The MC can become time-dependent if the driving forces exceed a certain value similar to buoyancy driven convection. Presently very few experimental results exist concerning the transition from steady to unsteady MC in liquid metals and semiconductor melts...." (3, p. (7)297).

References/Applicable Publications:

(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 107-108. (in German; post-flight)

(2) Input received from Principal Investigator G. Müller, August 1993.

(3) Rupp, R., Auerochs, A., Müller, G., Weyrich, C., and Leibenzeder, S.: Growth of GaAs Single Crystals by the Floating Zone Technique Under Microgravity. Adv. Space Res., Vol. 11, No. 7, 1991, pp. (7)297-(7)304. (discusses later TEXUS efforts; does not specifically mention this or the TEXUS 16 experiment)

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Co-Investigator(s): Tison, P. (2)
Affiliation(s): (1,2) Laboratoire d'Etude de la Solidification, Centre d'Etudes Nucléaires de Grenoble (CENG), Grenoble, France

Experiment Origin: France
Mission: MASER 2
Launch Date/Expt. Date: February 1988
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: Crystal Growth Furnace (CGF). (The CGF module contained four ellipsoidal mirror furnaces.)
Builder of Processing Facility: Swedish Space Corporation (SSC), Solna, Sweden

Experiment:
Semi-Confined Bridgman Growth of Ga Doped Ge

During this MASER 2 sounding rocket experiment, confined and semi-confined Bridgman growth of gallium doped germanium was investigated.

Both the confined and semi-confined sections of the experiment employed a 6 mm diameter rod of Ga doped Ge as the sample material. Each rod was maintained between silica supports and configured within a quartz ampoule. (The ampoules were sealed under 10^{-4} Pa of Ar.) The sample used for the semi-confined part of the experiment was configured in the ampoule such that it had a 7-mm long free surface section (before pulling was initiated) when melted. The sample used for the confined part of the experiment was configured within the ampoule such that it had no free surfaces when melted.

Using two of the four ellipsoidal furnaces housed within the Crystal Growth Furnace (CGF), the two samples were melted and then directionally solidified. Reportedly, "The thermal gradient in the molten zone was 55 ^{o}C/cm and the pulling rate was about 100 μm/s." (5, p. 188) It was expected that comparison of the samples would allow an assessment of the effect of thermocapillary flows on the macrosegregation of crystalline components.

Results of the Semi-confined Sample:
Reportedly, the semi-confined sample was processed as planned. "The analysis of the shape of the solidified floating zone of the partly covered sample showed that the wetting angle of the upper meniscus on the support increased from 130 to 150^{o} as the meniscus raised during the growth process. Grooves parallel to the sample axis can be observed on the lower - colder - part of the

floating zone which are attributed to the breaking of a native oxide layer on melting." (5, p. 188) "In the part solidified in contact with quartz, large cavities are observed. They are probably due to outgassing of the sample." (3, p. 5)

Longitudinal and radial segregations of the gallium were measured to determine the convective conditions within differing regions of the sample. The effective partition ratio derived from the longitudinal segregation of the semi-confined MASER sample was found to be higher (0.34) than the partition ratio derived from a similarly processed 1-g reference sample (0.15). "This indicated that convective mixing occurred in space, with a solute boundary layer even smaller than in conventional Bridgman configuration on Earth." (5, p. 188) Reportedly, this convection was weaker than expected, possibly because the governing surface tension driven flows "...were hampered by the oxide layer observed at the surface of the floated zone." (5, p. 188)

It appears that the radial segregation of the semi-confined MASER sample was found to be larger than the radial segregation of a similarly processed ground-based reference sample. Further, "A sharp maximum of the radial concentration profile could be observed at a well defined position all along the uncovered region of the [MASER] sample. This maximum, induced by convection, was not located on the sample axis, due to the asymmetry of the oxide skin, and due also to the local contribution to the mixing of a large bubble in the covered region." (5, p. 188)

Results of the Confined Sample:
The confined sample was not processed as planned. An "..early shut-off of the furnace occurred at the end of the heating phase... [and] outgassing caused separation of the liquid in two parts." (3, p. 5) Reportedly, "...segregations will be analysed[sic] in order to verify whether the contact between liquid and crucible was properly achieved." (3, p. 5)

<Note: A related experiment can be found under Carlberg, MASER 2 (this chapter). Reference (6) was not available at the time this experiment summary was prepared.>

Key Words: Crystal Growth From the Melt, Melt and Solidification, Float Zones, Directional Solidification, Bridgman Technique, Semi-Confined Bridgman Growth, Encapsulated Float Zones, Coated Float Zones, Coated Surfaces, Oxide Layer, Liquid Column Rupture, Binary Systems, Dopant, Free Surface, Free Surface Solidification, Surface Tension, Thermocapillary Convection, Marangoni Convection, Liquid/Gas Interface, Solid/Liquid Interface, Solidification Front Physics, Interface Shapes, Meniscus Shape, Wetting, Contact Angle, Thermal Gradient, Growth Rate, Liquid Film Rupture, Homogeneity, Segregation, Striations, Bubbles, Bubble Formation, Outgassing, Cavity, Sample Detachment From Crucible, Semiconductor Applications, Furnace Malfunction, Incomplete Sample Processing

Number of Samples: two
Sample Materials: germanium doped with gallium (Ge*Ga*)
Container Materials: quartz ampoules (Si*O*)

Experiment/Material Applications:
These investigations were performed to evaluate the potential for successful, low-gravity, float-zone crystal processing.

References/Applicable Publications:
(1) Zaar, J. and Änggard, K.: Maser and Its Effectiveness and Experimental Results. In In Space '87, Japan Space Utilization Promotion Center (JSUP), October 13-14, 1987, 32 pp. (preflight)

(2) Zaar, J. and Dreier, L: MASER II Final Report, RMLO/1-7, Swedish Space Corporation, August 30, 1988. (post-flight)

(3) Semi-Confined Bridgman Growth of Ga Doped Ge. In MASER II Final Report, RMLO/1-7 Swedish Space Corporation, August 30, 1988, Appendix 5, pp. 4-6. (post-flight)

(4) Input received from Experiment Investigator, September 1988.

(5) Semi-Confined Bridgman Growth of Ga-doped Ge. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 188-189. (post-flight)

(6) Camel, D. and Tison, P.: Semi-Confined Bridgman Growth of Ga-doped Ge in MASER 2. Proceedings VIIth European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-225, January 1990, pp. 63-68. (post-flight)

(7) Input received from Principal Investigator D. Camel, July 1993.

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CHAPTER 10

CRYSTAL GROWTH FROM VAPOR

Principal Investigator(s): Rubenstein, M. (1), Davidson, M. C. (2)
Co-Investigator(s): Unknown
Affiliation(s): (1) During Skylab: Westinghouse Electric Corporation, Monroeville, Pennsylvania, Currently: Deceased; (2) During Skylab: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama, Currently: SEMTEC, Madison, Alabama

Experiment Origin: USA
Mission: Was to fly on Skylab, SL-2, First Skylab Manned Mission
Launch Date/Expt Date: It appears that this experiment was dropped from the manifest late in pre-mission scheduling due to safety considerations (see Experiment summary below).
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels, located forward from the Multiple Docking Apparatus (MDA) Area, Skylab Manned Environment
Processing Facility: Furnace which contained three ampoules and used the M512 Materials Processing Facility heat sink cavity.
Builder of Processing Facility: Unknown

Experiment:
Gallium Arsenide Crystal Growth (M555)

When crystals are grown from metallic solutions on Earth buoyancy-driven convective flows induce crystalline defects. These defects are detrimental to the performance of the crystals and limit the usefulness of the material. In a low-gravity environment such convective flows are suppressed and crystals with improved properties should result.

The objective of the Skylab experiment was to produce single crystals of gallium arsenide which exhibited an exceptionally high chemical and crystalline perfection.

Prior to the Skylab mission, three specially constructed fused-quartz growth ampoules were prepared. Each ampoule contained (1) the source material (chunks of high purity GaAs), (2) a solvent, and (3) a single crystal GaAs seed. Two of the ampoules contained pure GaAs seeds; one ampoule contained a GaAs seed doped with silicon.

During the mission, the source material was to be dissolved in a liquid gallium melt at the hot end of the ampoule. The GaAs vapor was then expected to transport by diffusion down the ampoule and deposit on the seed. The three ampoules were prepared such that (1) a pure GaAs layer would be grown on a pure seed (ampoule 1), (2) a pure GaAs layer would be grown on a doped seed

(ampoule 2), and (3) a doped GaAs layer would be grown on a pure seed (ampoule 3).

"The three experiment ampoules were packaged in a cylindrical container (furnace), 10 cm (4 inches) diameter by 29 cm (11.5 inches) in length. The furnace was stored in the launch container... at all times except during the actual experiment operation.

"The launch container supplied the heated environment required to maintain the ampoule contents in the liquid state before and after the crystal growth process. The launch container had dimensions of 46 cm (18 inches) by 20 cm (8 inches) by 28 cm (11 inches) and weighed 9.1 kg (20 lb) with the furnace installed. An electrical connector was provided to interface with both the CM [Command Module] and MDA [Multiple docking adapter]. The container was launched in the CM, where 3 watts (at +28 VDC) of continuous power were required. After docking, the container was transferred to its storage position on the M512 [Materials Processing Facility] mounting panel..., and electrically connected to the AM Power Bus until experiment performance." (3, p. 5-40)

It was intended that the furnace would be removed from the launch container and inserted into the M512 chamber heat sink cavity. The furnace was then to be connected to the power source and experiment operation begun by activation of a control panel switch. The subsequent experiment time of 115 hours would not have required crew interaction.

The experiment hardware was scheduled to be launched on all four Skylab missions. However, the hardware was removed from the missions, prior to launch, for various reasons (see Reference (3), p. 5-40 for details).

Key Words: Crystal Growth From Vapor, Single Crystals, Seed Crystals, Binary Systems, Metallic Solutions, Crystal Homogeneity, Crystalline Defects, Dopant, Source Material, Solvent, Vaporization, Dissolution, Diffusion, Diffusive Mass Transfer, Heat and Mass Transfer, Vapor Transport, Vapor Deposition, Vapor/Solid Interface, Films, Film Growth, Film Microstructure, Buoyancy-Driven Convection, Semiconductor Applications, Electronic Materials, Sample Not Processed As Planned

Number of Samples: three
Sample Materials: gallium arsenide (Ga*As*)
Container Materials: fused quartz (Si*O*)

Experiment/Material Applications:
Crystals grown from metallic solutions (while under low-gravity conditions), are expected to have higher quality performance characteristics.

References/Applicable Publications:
(1) Rubenstein, M., Hopkins, R. H., and Kim, H. B.: Research Studies on Materials Processing in Space Experiment M512. TSLP Final Report, June 15, 1972-November 30, 1973, NASA CR-120418, 105 pp.

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab; post-flight)

(3) "Experiment M555-GaAs Crystal Growth". In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-39 - 5-42. (preflight objectives)

(4) "M512-Materials Processing Facility", In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, pp. 5-1 - 5-18. (processing facility)

(5) Gallium Aresenide Crystal Growth (M555). In Skylab Experiments, Vol. 3, Materials Science, produced by the Skylab Program and NASA's Education Programs Division in cooperation with the University of Colorado, May 1973. For sale by the Superintendent of Documents, U.S. Government Printing Office, pp. 17-19. (post-flight)

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Principal Investigator(s): Wiedemeier, H. (1)
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Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt. Date: September 1973 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Skylab Manned Environment
The experiment was configured in the Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area.
Processing Facility: Multipurpose Electric Furnace System (MEFS) (The MEFS was capable of accommodating three samples.)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Vapor Growth of II-VI Compounds (M556)

Under the best conditions, Chemical Vapor Transport (CVT) experiments produce well-defined single crystals. However, the actual mass transport of the chemical systems toward the condensation region is dependent on several factors including diffusion, buoyancy-driven convection, and ampoule pressure. Ground-based studies have indicated that habit and morphology of specific GeSe and GeTe chemical systems are significantly degraded by convective contributions.

This Skylab SL-3 experiment was the first in a series of investigations designed by Wiedemeier et al. to study vapor crystal growth. The overall objective of the experiment was to grow GeSe and GeTe crystals by the CVT method. Secondary objectives of the research included comparing ground-based and space-based (1) mass transport rates within the chemical systems, and (2) resultant crystalline morphology.

Prior to the mission, three flight ampoules were prepared to specifically investigate the role of the convection component in the overall transport of the system. The ampoules (and their source/transport-agent combination) were designated as systems 3A, 3B, and 3C. (See the **Sample Materials** section (below) for specific chemical compositions. The transfer agent fill-pressure varied in systems A, B, and C.) During ground-based investigations, the chemical system corresponding to system 3A exhibited a *high degree* of convection, the chemical system corresponding to system 3B exhibited a low degree of convection, and the chemical

system corresponding to system 3C exhibited a medium degree of convection.

The single (temperature) zone Skylab furnace called the Multipurpose Electric Furnace System (MEFS)) was converted into a double (temperature) zone furnace for the experiment by placing heat shields in the ampoules and furnace. Three evacuated, sealed ampoules could then be simultaneously subjected to a linear temperature gradient. At the hot end (520 ^{o}C), the solid source material (GeSe or GeTe) reacted with the GeI_4 transport agent. The gaseous products then migrated to the colder end of the ampoule (420 ^{o}C) where they condensed to form the resultant solid crystal.

The overall experiment timeline proceeded as follows: after the temperature gradient was achieved (2.75 hours), it was maintained (33 hours), and then terminated (allowing ambient cooling of the samples (12.5 hours)).

Post-flight, the Skylab-processed samples were compared to similarly Earth-processed samples. Reportedly, for the space samples, the mass flux from the source to the crystal condensation region (assuming only gas diffusion had occurred) was greater than anticipated. "For systems (A) and (C), the differences between observed and predicted mass fluxes... [were] about one order of magnitude. These observations... [indicated] the existence of other than gravity-driven convective components contributing to mass flux in a reactive solid-gas phase system. Thermodynamic and kinetic considerations... [suggested] that the additional transport modes... [were] related to the thermochemistry of the gas phase reaction and to secondary effects of the temperature gradient." (4, p. 241)

It was also reported that while ground-processed GeTe usually had poor crystalline structure, distorted surfaces, and hollow growth habits, the space-grown GeTe crystals "...have considerably more compact habits. The as-grown faces of these crystals show a higher degree of smoothness and crystalline perfection." (4, p. 242) Further, "The most pronounced difference in growth morphology was predicted and observed for GeSe (...3A)." (4, p. 243) While the ground-based sample contained dendritic growth (indicating the effects of convection on the crystal habit), the space-grown crystal had "...individual, well-developed single crystal platelets.... The space ampoule 3A... [contained] the largest GeSe single crystal grown under present experimental conditions." (4, p. 243)

Key Words: Crystal Growth From Vapor, Vapor Transport, Chemical Vapor Transport, Binary Systems, Single Crystals, Source Material, Transport Agent, Thermal Gradient, Vaporization, Sublimation, Condensation, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Passive Cooling, Vapor/Solid Interface, Vapor Deposition, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Dendritic Solidification, Dendrites, Platelet Habit, Reaction Kinetics, Gas Pressure, Electronic Materials, Semiconductor Applications

Number of Samples: three
Sample Materials: Sample 3A: 2.0 gm GeSe (source material), 14.28 mg GeI_4 per cm^3 tube volume (transport agent), 1.50 atm transport agent fill pressure; Sample 3B: 1.0 gm GeSe (source material), 1.28 mg per cm^3 GeI_4 (transport agent), 0.13 atm transport agent fill pressure; Sample 3C: 1.0 gm GeTe (source material), 7.14 mg GeI_4 per cm^3 (transport agent), 0.75 atm.
(Ge*Te*, Ge*Se*, Ge*I*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
The CVT crystal growth method is often used to produce single crystals used in high performance, electronic devices such as semiconductors. Because homogeneity and crystalline perfection are strongly dependent on the transport and condensation of the chemical system, the roles of these variables were investigated. It was expected that the space environment would produce a reduction of convective flow, contributing to superior crystalline quality.

GeSe and GeTe "...are presently of limited interest as electronic device materials... [although] their structural properties are favorable for detection of morphological changes." (4, p. 236)

References/Applicable Publications:
(1) Wiedemeier, H., Klaessig, F. C., Irene, E. A., and Wey, S. J.: Crystal Growth and Transport Rates of GeSe and GeTe in Microgravity Environment. Journal of Crystal Growth, Vol. 31, 1975, pp. 36-43. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) Naumann, R. J. and Herring, H. W.: Experiment M556, Crystal Growth by Vapor Transport. In Materials Processing in Space: Early Experiments, NASA SP-443, pp. 59-62. (post-flight)

(4) Wiedemeier, H., Klaessig, F. C., Wey, S. J., and Irene, E. A.: Vapor Growth of GeSe Single Crystals in Micro-gravity. In Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. 1, April 30-May 1, 1974, NASA Marshall Space Flight Center, Alabama, June 1974, pp. 235-257. (post-flight)

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(6) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(7) Experiment M556-Vapor Growth of IV-VI Compounds. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-56 - 5-57. (post-flight)

(8) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop. NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(9) Vapor Growth of IV-VI Compounds (M556). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-49. (post-flight)

(10) Wiedemeier, H.: Crystal Growth in Microgravity-An Overview. In Applications of Space Flight in Materials Science and Technology, proceedings from a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, issued September 1978, pp. 25-39. (post-flight)

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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: January 1974 (month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Skylab Manned Environment
The experiment was configured in the Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area.
Processing Facility: Multipurpose Electric Furnace System (MEFS) (The MEFS was capable of accommodating three samples.)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Vapor Growth of II-VI Compounds (M556)

This Skylab SL-4 experiment was the second in a series of investigations designed by Wiedemeier et al. to study vapor crystal growth (see Wiedemeier Skylab, SL-3). The overall objectives of the experiment were to (1) grow GeSe and GeTe crystals by the chemical vapor transport (CVT) method, (2) study the effects of low-gravity on vapor crystal production, and (3) determine the mass transport rates of the vapor system. A secondary objective of the experiment was to verify the increased mass transport rates observed during earlier SL-3 CVT processing (see Wiedemeier, SL-3).

As in SL-3, the three flight ampoules were prepared to specifically investigate the role of the convection component in the overall transport of the system. The ampoules contained the same source/transport agent combinations as on SL-3 and were designated as systems 5A, 5B, and 5C. (See the **Sample Materials** section (below) for specific chemical compositions. The transfer agent fill pressure varied in systems A, B, and C.)

During the SL-4 mission, the three evacuated, sealed ampoules were simultaneously subjected to a temperature gradient within the Skylab Multipurpose Furnace. While the SL-3 experiment employed a temperature gradient of 520 ^{o}C-420 ^{o}C, this SL-4 experiment employed a temperature gradient of 412 ^{o}C-346 ^{o}C. At the hot end, the solid source material (GeSe or GeTe) reacted with the GeI_4 transport agent. The gaseous products then migrated to the colder end of the ampoule where they condensed to

form the resultant solid crystal.

The overall experiment timeline (which was somewhat different from the SL-3 experiment) proceeded as follows: After the temperature gradient was achieved (1.5 hours), it was maintained (34 hours) and then terminated (allowing ambient cooling of the samples (7 hours)).

Post-flight, the Skylab-processed samples were compared to similarly-processed terrestrial samples. Analysis of the space samples indicated that because of the lower SL-4 temperature gradient "...a partial precipitation of transport agent occurred in ampoules 5A and 5C during transport. No precipitation of GeI_4 occurred in ampoule 5B. The resulting pressure conditions corresponded to a medium (GeSe, 5A and GeTe, 5C) and to a low (GeSe, 5B) convective contribution to the overall transport under ground-based conditions." (4, p. 238) Reportedly, "Despite the lower temperature gradient and the associated partial precipitation of transport agent for the SL-4 experiments, the flux data... [were] in good agreement with the overall pattern observed during the SL-3 mission." (4, p. 242)

When compared to the SL-3 results, the lower temperature gradient resulted in smaller transport rates and a reduction in mobility of the species on the surface of the crystal. Reportedly, the morphology of the crystals grown during SL-4 showed the same trends as the SL-3 samples when compared to their respective grown-based counterparts.

Key Words: Crystal Growth From Vapor, Vapor Transport, Chemical Vapor Transport, Binary Systems, Single Crystals, Source Material, Transport Agent, Thermal Gradient, Vaporization, Sublimation, Condensation, Precipitation, Reaction Kinetics, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Passive Cooling, Vapor/Solid Interface, Vapor Deposition, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Gas Pressure, Electronic Materials, Semiconductor Applications, Vacuum

Number of Samples: three

Sample Materials: Sample 5A: 2.0 gm GeSe (source material) and 14.28 mg GeI_4 per cm^3 tube volume (transport agent); Sample 5B: 1.0 gm GeSe (source material) and 1.28 mg GeI_4 per cm^3 (transport agent); Sample 5C: 1.0 gm GeTe (source material) and 7.14 mg GeI_4 per cm^3 (transport agent). <Note: the transport agent fill pressures were not specifically stated in the available references but were most likely the same as those of Wiedemeier's SL-3 experiments.>
(Ge*Te*, Ge*Se*, Ge*I*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
See Wiedemeier, Skylab, SL-3.

References/Applicable Publications:
(1) Wiedemeier, H., Klaessig, F. C., Irene, E. A., and Wey, S. J.: Crystal Growth and Transport Rates of GeSe and GeTe in Microgravity Environment. Journal of Crystal Growth, Vol. 31, 1975, pp. 36-43. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) Naumann, R. J. and Herring, H. W.: Experiment M556, Crystal Growth by Vapor Transport. In Materials Processing in Space: Early Experiments, NASA SP-443, pp. 59-62. (post-flight)

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(10) Naumann, R. J. and Mason, D.: Vapor Growth of IV-VI Compounds. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 20-21. (post-flight)

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Experiment Origin: USA
Mission: Apollo-Soyuz Test Project (ASTP)
Launch Date/Expt. Date: July 1975
Launched From: NASA Kennedy Space Center, Florida
Payload Type: ASTP Docking Module Payload
Processing Facility: Multipurpose Electric Furnace (gradient furnace) located in ASTP docking module
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Crystal Growth from the Vapor Phase (MA-085)

This Apollo-Soyuz Test Project (ASTP) experiment was the third in a series of investigations designed by Wiedemeier, et al. to study vapor crystal growth (see Wiedemeier Skylab SL-3 and SL-4). The overall objectives of the experiment were to (1) grow GeSeTe, GeSSe and GeS crystals by the chemical vapor transport (CVT) method, (2) study the effects of low gravity on vapor crystal production, and (3) determine the mass transport rates of the vapor system. A secondary objective of the experiment was to extend the investigations performed by Wiedemeier during SL-3 and SL-4 to examine more complex source/transport-agent systems processed in higher temperature gradients.

Prior to the mission, three flight ampoules were prepared. The ampoules were designated as systems A, B, and C. (See the **Sample Materials** section (below) for specific source/transport-agent chemical compositions.) Argon was added to system C to increase the ampoule pressure by a factor of three, and thus allow insight into the transport rates observed in the Skylab experiments.

During the mission, the three evacuated, sealed ampoules containing the source/transport components were simultaneously subjected to a nearly linear temperature gradient within the ASTP Multipurpose Electric Furnace. At the hot end (604 ^{o}C), the solid source material (GeSeTe, GeSSe, or GeS) reacted with the transport agent (GeI_4 or $GeCl_4$). The gaseous products migrated to the colder end of the ampoule (507 ^{o}C) where they condensed to form the resultant solid crystal. The overall experiment timeline proceeded as follows: After the temperature gradient was achieved (2.0 hours), it was maintained (16 hours), and then terminated. Cooling of the samples then took place (within 4 hours). (At some

point during this cooling time, a helium quench was used.)

Post-flight, the space-processed samples were compared to similarly processed Earth-based samples using a variety of techniques. Many results were detailed, some of which are presented here.

Reportedly, "...the lattice constants of the respective systems [space systems A, B and C compared to earth systems A, B and C]... [were] in excellent agreement. There... [was] no change in the orientation of predominant native faces of single crystals. Within the detection limits of X-ray diffraction techniques, there... [was] no measurable effect of microgravity on the crystallographic parameters of space-grown crystals." (2, pp. 475-476)

Laue X-ray diffraction transmission photographs of both the space and Earth-based systems indicated that both the space A and B systems had improved crystalline structure. For example, the ground-based crystal of system A had a "...high degree of strain and structural inhomogeneities..." (2, p. 476) whereas the space crystal indicated improved crystalline perfection. In system C, ground-based and space-based "...crystals grown in the presence of argon... [indicated] in both cases some degree of strain.... [C]rystals under these conditions... [were] considerably thinner than those of systems A and B." (2, p. 476)

Chemical homogeneity of the solid solutions (systems A and B) were also presented. It was noted that the distribution of Te in GeSeTe, and Se in GeSSe, was more homogeneous in the space crystals. For system A, the improvement was approximately one order of magnitude; the improvement was less for system B, but still measurable.

Mass transport rates were determined by comparing the mass of the resultant crystals to the remaining mass of the source materials. For system A, "...the microgravity flux... [was] lower than the normal gravity flux but approximately three times greater than predicted for a purely diffusion-type transport.... For system B, the normal gravity and microgravity fluxes... [were] the same, but the latter... [was] more than three times greater than the transport rate predicted by theory.... For system C, the normal gravity and microgravity fluxes... [were] the same within error limits and the latter... [was] nearly five times greater than predicted. A comparison of the individual flux data of systems B and C... [demonstrated] that the effect of argon on the mass transport rate... [was] the same under normal gravity and microgravity conditions." (2, p. 487)

It was concluded that Skylab and ASTP results were found to be in excellent agreement; thus, the possibility of producing higher quality crystals by vapor growth might be a space processing option.

Key Words: Crystal Growth From Vapor, Vapor Transport, Chemical Vapor Transport, Ternary Systems, Binary Systems, Single Crystals, Source Material, Transport Agent, Thermal Gradient, Vaporization, Sublimation, Condensation, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Vapor/Solid Interface, Vapor Deposition, Solid Solution, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Crystalline Strain, Surface Morphology, Quench Process, Gas Pressure, Electronic Materials, Semiconductor Applications

Number of Samples: three
Sample Materials: (System A) contained 1.50 gm $GeSe_{0.99}Te_{.01}$(source) and 61.8mg GeI_4 germanium tetraiodide (transport agent); (System B) contained 1.5 gm $GeS_{0.98}Se_{0.02}$ (source) and 59.1 mg $GeCl_4$ (transport agent); (System C) contained 1.50 gm GeS (source) and a mixture of $GeCl_4$ and 1.91 atm argon (transport agent)
(Ge*Se*Te*, Ge*I*, Ge*S*Se*, Ge*S*, Ge*Cl*, Ar*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
See Wiedemeier, SL-3.

References/Applicable Publications:
(1) Wiedemeier, H., Sadeek, H., Klaessig, F. C., Norek, M., and Santandrea, R.: Crystal Growth from the Vapor Phase, Experiment MA-085. Final Report, NASA-CR-161965.

(2) Wiedemeier, H., Sadeek, H., Klaessig, F. C., Norek, M., and Santandrea, R.: Crystal Growth from the Vapor Phase Experiment MA-085. In Apollo-Soyuz Test Project Summary Science Report, Vol. I, NASA SP-412, 1977, pp. 471-489.

(3) Boese, A., Mchugh, J., and Seidensticker, R.: Multipurpose Electric Furnace. In Apollo-Soyuz Test Project Summary Science Report, Vol. I, NASA SP-412, 1977, pp. 353-365. (post-flight)

(4) Wiedemeier, H., Sadeek, H., Klaessig, F. C., Norek, M., and Santandrea, R.: Crystal Growth from the Vapor Phase; Experiment MA-085. In Apollo-Soyuz Test Project-Composite MSFC Final Science Report, NASA TM X-73360, January 1977, pp. VII-1 - VII-40. (post-flight)

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(6) Naumann, R. J. and Mason, E. D.: Crystal Growth from the Vapor Phase. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, p. 64, August 1979. (post-flight)

(7) Naumann, R. J. and Herring, H. W.: Experiment MA 085, Crystal Growth from the Vapor Phase. In Materials Processing in Space: Early Experiments, NASA SP-443. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #7, STS-007 (STS 31-C, Challenger)
Launch Date/Expt. Date: June 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Payload Bay OSTA-2 Payload Pallet Platform, Materials Experiment Assembly (MEA-A1)
Processing Facility: Three-zone electric furnace (capable of accommodating two samples)
Builder of Processing Facility: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Vapor Growth of Alloy Type Semiconductor Crystals

This STS-007 experiment was the fourth in a series of investigations designed by Wiedemeier et al. to study vapor crystal growth (see Wiedemeier Skylab SL-3, SL-4, ASTP). The overall objectives of the experiment were to (1) grow GeSe crystals by the physical vapor transport (PVT) method, (2) study the effects of low gravity on vapor crystal production, and (3) determine the mass transport rates of the vapor system.

Experimental results from Wiedemeier's Skylab and ASTP studies indicated that while (1) the chemical and structural microhomogeneity of the Skylab and ASTP chemical vapor transport (CVT) grown crystals were of superior quality than similarly processed terrestrial crystals, (2) low-gravity CVT rates were unexpectedly higher than rates predicted by pure-diffusion-type transport. Results further indicated that "...the discrepancy between observed and expected mass transport rates in space... [had increased] with increasing transport rate pressure." (3, p. 121) Thus, it had been speculated that the additional mass flux might be attributed to the thermal-chemical effects of homogeneous gas-phase reactions.

In an effort to reduce the complexities of the multi-component, multi-reaction CVT system (and further investigate the cause of the additional mass flux), the transport agent was removed for these STS experiments and replaced with the inert gas xenon. In such a physical vapor transport (PVT) system, chemical phase reactions are essentially absent.

During the mission, two ampoules containing the source/buffer components were simultaneously subjected to a temperature gradient of 600 ^{o}C-500 ^{o}C within a three-zone electric furnace. The first ampoule (designated as MW11) contained 2.0 gm of polycrystalline GeSe source material and 4 atm of xenon. In this ampoule, transport was allowed for a duration of 18 hours. The second ampoule (designated as MW14) contained a similar amount of GeSe, but 8 atm of xenon. In this ampoule, transport was allowed for a duration of 30 hours. During the processing, the GeSe source was heated to sublimation (at the hot end) and diffused through the buffer xenon gas to the opposite end of the ampoule. The vapor, now at a lower temperature, condensed to form the resulting solid crystal.

Post-flight, the space-processed samples were compared to similarly processed 1-g samples using a variety of techniques. Many results and conclusions were presented. Among them were:

(1) "The mass transport rates of GeSe observed in [the] microgravity environment are in close agreement with theoretically predicted values for diffusion limited mass transport....

"[2] [The above result] ...supports the earlier proposed hypothesis for the interpretation of flux anomalies observed in previous space experiments....

"[3] ...the STS flight experiments led to the observation of unexpected crystal growth phenomena. The largest GeSe single crystals obtained in microgravity grew in the... [ampoule] without direct wall contact which could suggest homogeneous nucleation....

"[4] The space grown crystals are much larger and have considerable improved surface and bulk morphologies relative to corresponding ground control specimens." (1, p. 1015, abstract)

Key Words: Crystal Growth From Vapor, Vapor Transport, Physical Vapor Transport, Binary Systems, Single Crystals, Source Material, Thermal Gradient, Vaporization, Sublimation, Condensation, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Vapor/Solid Interface, Vapor Deposition, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Nucleation, Gas Pressure, Sample Detachment From Crucible, Electronic Materials, Semiconductor Applications

Number of Samples: two
Sample Materials: Both samples employed 2.0 g polycrystalline GeSe as the source material. The first experiment (MW11) contained 4 atm xenon, while the second experiment (MW14) contained 8 atm Xenon. (Xenon used as a buffer gas.)
(Ge*Se*, Xe*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
See Wiedemeier, Skylab SL-3.

References/Applicable Publications:
(1) Wiedemeier, H., Trivedi, S. B., Zhong, X. R., and Whiteside, R. C.: Crystal Growth and Transport Rates Of the GeSe-Xenon System Under Microgravity Conditions. Journal Of Electrochemical Society, Vol. 133, May 1986, pp. 1015-1021. (post-flight)

(2) STS-7 Seventh Space Shuttle Mission, NASA Press Kit, June 1983, pp. 48-49. (preflight)

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Experiment Origin: USA
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Payload Bay, Materials Experiment Assembly (MEA-A2)
Processing Facility: Gradient General Purpose Rocket Furnace (G-GPRF)
Builder of Processing Facility: Unknown, possibly, National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

Experiment:
Vapor Growth of Alloy-Type Crystals (ME-GPRF 4)

This Spacelab D1 experiment was the fifth in a series of experiments designed by Wiedemeier et al. to study vapor crystal growth (see Wiedemeier, Skylab SL-3, SL-4, ASTP, STS-007). The overall objectives of this experiment were to (1) grow GeSe crystals by the Physical Vapor Transport (PVT) method, (2) study the effects of low gravity on vapor crystal production, and (3) determine the mass transport rates of the vapor system.

Experimental results from Wiedemeier's earlier STS-007 investigation indicated that while (1) low-g mass transport rates of the GeSe-xenon system were similar to those transport rates defined by a purely diffusive transport, (2) unexpected crystal growth was observed. Therefore, the major objectives of this experiment were to (1) verify the STS-007 results and (2) establish a flux versus pressure curve for the low-gravity processed samples.

Prior to the Spacelab D1 mission, two flight ampoules were prepared. Similarly to the STS-007 experiments, 2.0 gm of polycrystalline GeSe was used as the source material in both D1 ampoules. The major difference between the two shuttle experiments was that the ampoules contained different xenon pressures: D1 ampoule 1 contained 2 atm of xenon and D1 ampoule 2 contained 6 atm of xenon (compared to 4 atm and 8 atm of xenon in ampoules on STS-007). These differing D1 pressures were chosen such that two more data points could be attained for a low-gravity flux vs. pressure curve.

During the mission, the ampoules were subjected to a temperature gradient of 600 ^{o}C-500 ^{o}C within the Gradient General Purpose Rocket Furnace. (This gradient was the same as that employed during the STS-007 mission.) The GeSe source was heated to sublimation (at the hot end) and diffused through the buffer xenon gas to the opposite end of the ampoule. The vapor, now at a lower temperature condensed to form the resulting solid crystal.

Post-flight evaluation of the samples revealed that "The deposition regions of both ampoules... [had] a similar appearance of those of the preceding STS-7 experiments. Besides the deposition and growth of GeSe crystals on the ampoule wall, there ...[were] several large, single crystalline GeSe platelets with lateral dimensions much greater than those of crystals on the wall and obtained on [the] ground.... Several of these larger crystals... [were] not in direct contact with the ampoule wall, but ... [were] supported by thin crystals attached to the wall. The surfaces of the large crystals... [were] highly reflective and nearly mirror smooth. These preliminary observations of the D1 experiments... [confirmed] the crystal growth pattern, sizes, and surface morphology of the previous space-grown crystals (STS-7...) for different pressures and... [re-emphasized] the question concerning nucleation phenomena in microgravity." (2, p. 377)

It was also noted that "The mass transport rates... [agreed] within error limits with diffusion-controlled mass fluxes. With these two D1 experiments, a reliable mass flux versus pressure curve for the physical vapor transport of GeSe in microgravity has been established." (1, p. 89)

Key Words: Crystal Growth From Vapor, Vapor Transport, Physical Vapor Transport, Vapor Deposition, Binary Systems, Single Crystals, Source Material, Thermal Gradient, Vaporization, Sublimation, Condensation, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Vapor/Solid Interface, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Nucleation, Platelet Habit, Gas Pressure, Sample Detachment From Crucible, Electronic Materials, Semiconductor Applications

Number of Samples: two

Sample Materials: Both ampoules had 2.0 g polycrystalline GeSe as source material. The first ampoule contained 2 atm xenon, while the second ampoule contained 6 atm xenon (xenon used as a buffer gas).
(Ge*Se*, Xe*)
Container Materials: fused silica
(Si*O*)

Experiment/Material Applications:
See Wiedemeier, Skylab SL-3.

References/Applicable Publications:
(1) Wiedemeier, H. and Triveldi, S. B.: Physical Vapor Transport and Crystal Growth of the GeSe-Xenon System in Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 88-89. (abstract only; post-flight)

(2) Wiedemeier, H. and Triveldi, S. B.: Initial Observations of GeSe-Xenon Transport Experiments Performed on the D1 Space Flight. Natur wissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 376-377. (post-flight)

(3) Wiedemeier, H.: Vapor Growth of Alloy-Type Semiconductor Crystals. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 124-125. (preflight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements)

(5) Materials Processing Experiments in Space: MEA-A2 Payload. Brochure available from Application Payload Projects NASA/MSFC, Huntsville, Alabama. (MEA; preflight)

(6) General Purpose Rocket Furnace. In Microgravity Science and Applications Experiment Apparatus and Facilities, document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 3-4. (processing facility)

(7) Naumann, R. J.: Microgravity Science and Applications. In: In Space 87, Japan Space Utilization Promotion Center (JSUP), pp. 24-25. (post-flight)

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Co-Investigator(s): Agawa, T. (2), Oda, H. (3) (Originators of Experiment Concept)
Affiliation(s): (1) During STS-006: Asahi Shimbun (Morning Sun Newspaper Company), Tokyo, Japan, Currently: Deceased; (2-3) During Launch: Japanese High School Students, Currently: Unknown

Experiment Origin: Japan
Mission: STS Launch #6, STS-006 (STS 31-B, Challenger)
Launch Date/Expt. Date: April 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) Canister G-005
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-005: Nippon Electric Company (NEC), Yokohama, Japan/The Asahi Shimbun Newspaper, Tokyo, Japan
Processing Facility: Two identical subsystems were designed to produce and observe the growth of snow crystals. Each subsystem was configured with (1) a cold chamber, (2) two high resolution video recorders, (3) a sequence control unit, and (4) a thermal control unit.
Builder of Processing Facility: Nippon Electric Company (NEC), Yokohama, Japan

Experiment:
Artificial Snowflake Crystal Experiment

In 1936, the first artificial snow crystals were produced by the Japanese physicist Ukichiro Nakaya. Almost 50 years later during this STS Get Away Special (GAS) experiment, Japanese investigators attempted the production of artificial snow in the low-gravity environment. Scientists speculated that space-produced snow crystals would be highly symmetrical or even spherical.

The space experiment was the first in a series of investigations designed by Kimura et al. to study the production of snow crystals in a low-gravity environment. The experiment concept was originally proposed by two Japanese High School students (listed above under the Co-Investigator designation). The experiment hardware, which was developed and manufactured within 8 months, was configured to produce snow crystals grown in air (not on the surface of an object).

The payload consisted of two identical subsystems, which allowed the observation of the crystalline production. Each subsystem was configured with (1) a small, lightweight, cold chamber (requiring low electrical power), (2) two compact high resolution video recorders (one with low power optics and one with high

power optics), (3) a sequence control unit, and (4) a thermal control unit. The power for the experiments was provided by Ni-Cd batteries.

Snow crystals were to be grown and observed in the cold chamber. The chamber was designed to (1) obtain and maintain the desired temperature environment, (2) continuously supply water vapor (to grow snow crystals), (3) supply small particles of silver iodine (to act as seeds for the snow crystals), and (4) illuminate the snow crystals (so the growth could be recorded).

The overall payload design also had to (1) provide additional cooling of the cold chamber (because of heat generated during the experiment and the insufficiency of available radiation cooling), (2) insure that water used in the snow production did not scatter during shuttle launch (because of high accelerations of varying frequencies), and (3) insure the water did not evaporate before the experiment was initiated.

Prior to the shuttle launch, approximately 15 grams of water were stored in a small, porous, sintered-metal container. The container lid was designed to prevent the stored water from evaporating or scattering before the experiment began.

Three days after STS-006 was launched, the experiment was activated via a signal from an STS astronaut. Within 5 minutes of activation, a command was sent by an astronaut to open the lid of the water container.

Reportedly, "The water temperature in the porous sintered metal container... [was to be] raised by an internal electric heater. This heater... [was] to maintain the water temperature at 20-30 degrees centigrade and thus supply water vapor continuously to the cold chamber. A very small heating element made of platinum... [was to be] activated in the cold chamber and... [was to warm] a few milligrams of silver iodine. The... iodine on the element sublimates, forming small particles that act as seeds for the snow crystals." (7, p. 5) The cold chamber was to be maintained at a temperature of -15 ^{o}C via semiconductor thermo-modules (cooling units).

"The growth forms of the snow crystals depend on the environmental temperature and the mass of water vapor supplied (in other words, supersaturation of surrounding water vapor.).... Predicting that the diffusion of the water becomes very small under microgravity, the mass of water vapor [was to be] made as large as possible." (7, p. 5)

During the mission, each subsystem performed two such snow experiments. The experiments each took 1 hour to complete, and were performed in sequence. (Thus, the total experiment time was 4 hours.)

Post-flight analysis of the video tapes appeared to indicate that the experimental apparatus functioned as expected. However, the expected crystal growth was not observed. "The data recorded on the audio channels of the video tapes revealed that the Experimental System was exposed to lower temperatures than anticipated in the cargo bay of the Space Shuttle." (7, p. 8) It appears that during the first snow experiment, the temperature inside the GAS canister was -7 ^{o}C. "Thus, the water in the container was frozen at the start of the experiment. The heater inside the container was of only 1 watt, so the temperature of the water could not be warmed up to 25 degrees centigrade. The experiment was repeated... four times but even at the last experiment the temperature of the water was about 7 degrees centigrade."

"It was inferred that, as a result, the... [amount] of the water vapor generated was small. Thus snow crystals were not formed. And it is also suspected that the lack of thermal convection under weightlessness prevented the water vapor... [from traveling the] 60 mm distance from the exit of the water container to the field of the view of the observation window." (2, p. 34)

The experiment was modified to address the anomaly and reflown onboard STS-008 (see Kimura, STS-008).

Ground-based experiments using the apparatus successfully produced snow crystals. During these experiments, a few rabbit hairs were placed across the chamber to catch and suspend the snow crystals that, because of gravity, fell to the bottom of the cold chamber. The diameters of the crystals varied from a few micrometers to several hundred micrometers. The video recorders captured the growth process of the crystals as well as (1) the temperatures of the cold chamber, and (2) the water supplying the vapor.

Key Words: Crystal Growth From Vapor, Vapor Transport, Snow Crystals, Spherical Crystals, Ice, Freezing, Vaporization, Sublimation, Seed Crystals, Supersaturation, Diffusion, Thermal Convection, Vapor/Solid Interface, Thermal Environment More Extreme Than Predicted, Processing Difficulties, Absence of Buoyancy Forces (Detrimental)

Number of Samples: Two experimental chambers were employed.
Sample Materials: Water vapor (silver iodide was used as seed crystals).
(H*O*, Ag*I*)
Container Materials: Not applicable. (The two cooling chambers were made from copper.)
(Cu*)

Experiment/Material Applications:
An example of the applications of this research was cited in Reference (7). Reportedly, "...the observation and recording techniques used in the Experimental System can be applied to observe and record the manufacturing process of alloys, semiconductors, and medicines under microgravity influence." (7, p. 8) Reference (6) indicated that "The experiment is expected to contribute to crystallography, especially the crystal growth of... [semiconductors] or other materials from a vapor source." (6, p. 41)

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS-6, JSC-17645 Annex STS-6, December 3, 1982. (very short description)

(2) Kimura, S., Oka, A., Taki, M., Kuwano, R., Ono, H., Nagura, R., Narimatsu, Y., Tanii, J., and Kamimiyata, Y.: Crystal Growth of Artificial Snow. In NASA Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, August 1-2, 1984, pp. 33-36, NASA CP-2324. (post-flight)

(3) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA GAS Team, 1989, p. 17. (post-flight, short description)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(5) "First Snow in Space--- A Space Shuttle Experiment," Asahi Evening News [Japan], Special Supplement.

(6) "STS-6 Getaway Specials," NASA News, NASA GSFC, November 24, 1982.

(7) Kimura, S., Shibata, T., Oka, A., Kuwano, R., Ono, H., Nagura, R., Narimatsu, Y., and Tanii, J.: Experimental System to Produce Artificial Snow on the STS-6. 34th Congress of the International Astronautical Federation, October 10-15, 1983, Budapest, Hungary, IAF Paper Number IAF-83-162. (post-flight)

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Co-Investigator(s): Agawa, T. (2), Oda, H. (3) (Originators of Experiment Concept)
Affiliation(s): (1) During STS-008: Asahi Shimbun (Morning Sun Newspaper Company), Tokyo, Japan, Currently: Deceased; (2-3) During Launch: Japanese High School Students, Currently: Unknown

Experiment Origin: Japan
Mission: STS Launch #8, STS-008 (STS 31-D, Challenger)
Launch Date/Expt. Date: August 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
NASA Get Away Special (GAS) Canister G-475
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-475: Nippon Electric Company (NEC), Yokohama, Japan/The Asahi Shimbun Newspaper, Tokyo, Japan
Processing Facility: The processing facility used during STS-006 (GAS G-005) was reused on this mission but was modified to include (1) 4.5 watt heaters to insure vaporization of experiment fluid (water) and (2) a fan to observe the effect of air flow on the snow.
Builder of Processing Facility: Nippon Electric Company (NEC), Yokohama, Japan

Experiment:
Artificial Snowflake Crystal Experiment

This experiment was the second in a series of investigations designed by Kimura et al. to study the production of snow crystals in the low-gravity environment (see Kimura, STS-006). Scientists had speculated that space-produced snow crystals would be highly symmetrical or even spherical.

The payload consisted of two identical subsystems which allowed the observation of crystalline production. These subsystems were very similar to those employed during the earlier GAS experiment. Reportedly, "The heart of the device for the experiments is two copper boxes, each 4 cm in both depth and width and 10 cm in height. On one side of each copper box, semiconductor thermo-modules (cooling units) are attached. The thermo-modules can lower the temperature in the box to -15 degrees centigrade in about 10 minutes. On the top of each copper box is a water container made of porous sintered metal which is similar to a sponge. In the water container is an electric heater to vaporize the water at about 25 degrees centigrade...."

"[Just below where the water vapor is formed]... there is a heater to sublimate silver-iodine, fine particles of which serve as seeds for snow crystals."

"There is... [an] observation window on the other side of the copper box, through which the snow crystals are observed with TV cameras and recorded onto video-tapes." (1, p. 33)

Four strands of rabbit hair, which were stretched vertically across the cold chamber, were used to capture the snow crystals for observation. Similarly, a piece of copper wire configured horizontally across the cold chamber was used to study the formation of frost. <Note: It is unclear if the earlier STS-006 version of this experiment employed the rabbit hairs and wire. Only ground-based experiments appear to have incorporated the hairs at the time of the STS-006 investigations (see Kimura, STS-006).>

Because snow crystals were not observed during the earlier STS-006 GAS experiment, two small improvements were made to the payload for this STS-008 flight:

(1) The replacement of the water vaporization heaters.
During STS-006, a 1 watt heater had been used to vaporize the water in each of the subsystems. Since the shuttle environment was colder than expected, this heater could not heat the water to the desired 25 ^{o}C-30 ^{o}C temperature. Therefore, these heaters were replaced with 4.5 watt heaters.

(2) Addition of a 1.5 cm diameter blower-type fan. In the low-gravity environment, convective flows within the chamber are reduced. It was suspected that during STS-006, this reduction was responsible for the inefficient transport of the water vapor (if it had indeed been produced) into the field of view of the cameras. Therefore, a 1.5 cm diameter blower-type fan was integrated into each of the subsystems to move the gas in the cold chambers.

During the STS-008 mission, each subsystem performed two snow experiments. The experiments were performed in sequence. (Thus a total of four experiments were performed.)

A document released prior to the shuttle launch outlined the expected fan operation sequences. "The fan will be changed in every snow-making experiment.... In the first experiment, the fan will be activated for the first third of the time. In the second experiment, the fan will be turned on from the beginning to the end of the experiment. In the third, the fan will be activated in the latter half, and in the final one, it will be on just for a short time at the beginning and a short time at the end of the experiment." (9, p. 40) <Note: It was unclear if this

exact fan sequence was realized during the experiments.>

Post-flight analysis of 6 hours and 50 minutes of space video tape indicated that the experiment was successful and that snow had been produced. Reportedly, during the second and third experiments (experiments in which the fan was activated to create the artificial breeze "during most of the experimental time") "...hexagonal and irregularly shaped snow crystals were formed on the rabbit hairs.... The results of these two experiments were just... [the] same as the results of the experiments which were carried out hundreds of times on the ground." (1, p. 35)

However, during the two other experiments, very different results were observed. In the first experiment, the fan was not activated during the initial 30 minutes of the experiment. <Note: It is not clear how long the fan was activated after this 30-minute time.> In the second experiment, the fan was activated for 5 minutes when the silver-iodine was sublimated. It then appears that the fan was turned off for the following 50 minutes. <Note: No further details of the experiments were described (for example, the total length of the experiments).> It appears that during both of these experiments, no snow came into the view of the cameras when the fan was not activated. However, when the fan was activated, nearly spherical, artificial snow crystals (formed presumably under near-weightlessness) were moved into view of the TV cameras by the fan breeze. "The diameter of the largest crystal was about 3 mm."

"One crystal traveled the field of... view from left to right and finally collide[d] with a rabbit hair. At the collision, the shape of the sphere was not changed. Thus, it was confirmed that the sphere was not a water droplet but a snow crystal." (1, p. 36)

Key Words: Crystal Growth From Vapor, Vapor Transport, Snow Crystals, Frost, Ice, Freezing, Vaporization, Sublimation, Seed Crystals, Supersaturation, Diffusion, Mass Transfer, Drops, Vapor/Solid Interface, Crystal Morphology, Spherical Crystals, Air Fan

Number of Samples: Two experimental chambers were employed.
Sample Materials: Water vapor (silver iodide was used as seed crystals.) The snow crystals were caught on four strands of rabbit hair configured in each chamber.
*(H*O*, Ag*I*)*

Container Materials: Not applicable. (The two cooling chambers were made of copper.)
(Cu*)

Experiment/Material Applications:
See Kimura, STS-006.

References/Applicable Publications:
(1) Kimura, S., Oka, A., Taki, M., Kuwano, R., Ono, H., Nagura, R., Narimatsu, Y., Tanii, J., and Kamimiyata, Y.: Crystal Growth of Artificial Snow. In NASA Goddard Space Flight Center Get Away Special Experimenter's Symposium, NASA CP-2324, 1984, pp. 33-36. (post-flight)

(2) Cargo Systems Manual: GAS Annex for STS-8. JSC-17656 Annex STS-8, July 15, 1983. (short description; preflight)

(3) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team, 1989, p. 17. (post-flight; short description)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(5) Four Get Away Special Experiments to Fly on STS-8 Mission. Goddard Space Flight Center, July 12, 1983.

(6) Getaway Special Payload for STS-8: Artificial Snow Crystal Experiment. NASA News, NASA Goddard Space Flight Center, July 12, 1983.

(7) Kolcum, E. H.: Eighth Space Shuttle Mission to Orbit Scientific Payloads. Aviation Week and Space Technology, August 22, 1983.

(8) First Snow in Space-- A Space Shuttle Experiment. Asahi Evening News [Japan], Special Supplement, October 24, 1983.

(9) STS-8 Eighth Space Shuttle Mission Press Kit, August 1983, pp. 39-40. (preflight)

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Co-Investigator(s): None
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Experiment Origin: France
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack
Processing Facility: One, two-zone, copper-water heat pipe furnace. The furnace was able to accommodate three experiment cartridges.
Builder of Processing Facility: L. E. P. Laboratory, Paris, France

Experiment:
Crystal Growth of Mercury Iodide by Physical Vapor Transport 1ES338

On Earth, gravity-driven convection (which occurs during vapor crystal growth) can result in unstable fluid flow at the crystal growth interface. The cystalline "...nucleation process is influenced by convection, even at pressures as low as 10^{-2} Torr... and [by] low mass Rayleigh numbers." (4, p. 440) If gravity-induced detrimental flows could be reduced in the space environment, higher quality crystals might be grown.

This Spacelab 1 experiment was the first in a series of investigations designed by Cadoret et al. to study the low-gravity, physical vapor transport (PVT) growth of mercuric iodide crystals.

It appears that three experiment cartridges resided in a two-zone heat pipe furnace during the mission. <Note: The specific HgI_2 systems in each of these cartridges were not detailed. However, it appears that at least one of the cartridges contained a HgI_2 system plus 10^{-2} Torr argon and another of the cartridges contained a HgI_2 system plus styrene.> The furnace was divided into two stabilized isothermal zones (a hot and cold zone) separated by a small temperature gradient. During the experiment, the material source was vaporized at the hot end of each cartridge. The resulting vapor was then deposited at the cold end of each cartridge and subsequent crystallization was realized.

<Note: Although Reference (4) discusses post-flight experiment results, it is difficult to distinguish between ground-based vs. space-based research. Further, the discussion is very short and important results are not clearly stated. Thus, the discussion (as presented in Reference (4)) is reproduced here almost in its entirety:>

It was briefly reported that (1) "The number of nuclei is much smaller in space experiments than on Earth... [and (2) the mass transport rate] decreases by a factor of 1.5 in space experiments." (4, p. 441)

"What is certainly encouraging, is that appreciable nucleation control could be achieved at these experiments. A large single crystal (compared to the time available) could be grown with with dimensions 4.5 X 4.5 X 3.5 mm^3.

"In other ampoules, Cadoret and Brisson have put 10^{-1} Torr argon, in addition to the HgJ_2[sic] charge. Both on earth and space more nuclei appear, leading to the assumption that adsorption or Ar on the ampoule walls may lead to additional nucleation centres.

"A third pair of ampoules was loaded with HgJ_2[sic] + styrene, an agent well known to lead to the formation of thin platelets of HgJ_2[sic], by poisoning the growth in certain lattice directions... with these experiments it was shown impressively that nucleation is strongly reduced in space. Of course, compared with the Ar experiments, the number of nuclei in the presence of the reactive impurity styrene increased appreciably." (4, p. 441)

<Note: When Principal Investigator R. Cadoret reviewed this experiment summary in October 1993 and sent back a brief letter highlighting his experiments, he wrote that he had enclosed a publication with the letter which detailed the Spacelab 1 experiment. However, no publication was found in the returned envelope, although Cadoret referred to the publication several times in his letter. Therefore, further details of this experiment are still unclear. Briefly in his letter he mentioned that it was concluded that "...the influence of gravity observed in SL1 [the Spacelab 1 experiment] on nucleation distribution, could influence crystal growth without changing the overall transport...."

"As a conclusion the nucleation SL1 experiments led us to suspect an effect of gravity on the growth of HgI_2 at low pressure.">

No further information describing the experimental results of this experiment could be located at this time. <Note: Neither Reference (7) nor (8) could be located for review.>

<Note: Cadoret also noted that the Spacelab 1 (and later Spacelab 3) results led to a new experiment configuration which was flown on the recent STS International Microgravity Laboratory 1 (IML-1) mission. (The IML-1 results are beyond the scope of this NASA Technical Memorandum (TM). These results will be published in a later version of the TM.)>

Key Words: Crystal Growth From Vapor, Vapor Transport, Physical Vapor Transport, Vapor Deposition, Binary Systems, Single Crystals, Source Material, Thermal Gradient, Vaporization, Sublimation, Condensation, Supersaturation, Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Vapor/Solid Interface, Interface Stability, Solidification Front Physics, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Impurities, Nucleation, Spurious Nucleation, Platelet Habit, Gas Pressure, Crucible Effects, Gamma Ray Detectors, Electronic Materials, Semiconductor Applications, Heat Pipes

Number of Samples: three
Sample Materials: mercury-iodide, HgI_2
<Note: Although it is not clearly stated in the available references, it appears that the first experiment cartridge contained an HgI_2 system, the second experiment cartridge contained an HgI_2 system plus 10^{-2} Torr argon, and the third experiment cartridge contained an HgI_2 system plus styrene.>
(Hg*I*)
Container Materials: unknown, possibly quartz
(Si*O*)

Experiment/Material Applications:
HgI_2 crystals are grown for use as low energy gamma-ray detectors.

HgI_2 was selected for the experiments "...because its nucleation is very sensitive to local supersaturation[;] this sensitivity is demonstrated by the difficulty of controlling the process in avoiding spurious nucleation." (2, p. 198)

References/Applicable Publications:

(1) Perrier, G., Belouet, C., Omaly, J., and Cadoret, R.: Vapour Growth of HgI_2 in Sealed Ampoules. In Space Research XIX, Proceedings of the Open Meetings of Working Groups on Physical Sciences, Innsbruck Austria, May 29-June 10, 1978, Oxford Pergamon Press Ltd., 1979, pp. 531-534. (preflight)

(2) Presenti, P.: Orientation and Perspectives of the French Materials Science in Space Programme. 30th International Astronautical Federation, International Astronautical Congress, Munich, West Germany, September 17-22, 1979, specifically pp. 197-198 (IAF Paper 79-59). (preflight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab 1)

(4) Kaldis, E.: Crystal Growth from Solutions and Nucleation from the Vapour Phase. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 439-441. (ground-based and post-flight experiments)

(5) Crystal Growth of Mercury Iodide by Physical Vapor Transport. In NASA/ESA Spacelab 1 Experiments, Marshall Space Flight Center 15M983, p. 14. (preflight)

(6) Input received from Experiment Investigator, December 1989 and October 1993.

(7) Cadoret, R. and Brisson, P.: The Effect of Gravity on Nucleation, Growth and Physical Transport of HgI_2. (space results/CNES-1984)

(8) Brisson, P., Magnan, A., and Cadoret, R.: First Results for HgI_2 Spacelab Experiments SL1 and SL3. (space results/CNES-1987; post-flight)

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Co-Investigator(s): None
Affiliation(s): (1,2) During STS 51-B: Université de Clermont-Ferrand, Aubière, France; (1) Currently: Université Blaise-Pascal, Laboratoire de Physique des Milieux Condensés, Aubière, France; (2) Currently: ESA/ESTEC, Noordwijk, The Netherlands

Experiment Origin: France
Mission: STS Launch #17, STS-024 (STS 51-B, Spacelab 3: Challenger)
Launch Date/Expt. Date: April 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack
Processing Facility: It appears that one, two-zone, copper-water heat pipe furnace was employed. Each zone was configured with two temperature monitoring sensors. (The furnace was the same as that employed during Cadoret's experiment on Spacelab 1.)
Builder of Processing Facility: L.E.P. Laboratory, Paris, France

Experiment:
Mercuric Iodide Crystal Growth (MICG)

This Spacelab 3 experiment was the second in a series of investigations designed by Cadoret et al. to study the low-gravity physical vapor transport (PVT) growth of mercuric iodide crystals (see Cadoret, Spacelab 1). The specific objective of the experiment was to focus "...on the location and number of nucleation sites as a function of temperature distribution and partial pressure of an inert host gas (argon)." (1, p. 4)

During the mission, two separate experiments were performed in a two-zone heat pipe furnace. The furnace (which had been previously employed on Spacelab 1) operated during the entire mission and was highly automated. During each of the two experiments, three mercuric-iodide samples were processed by the PVT method. During this processing (1) the samples were vaporized at the hot end of the zone furnace, (2) the resulting Stefan flow transported the vapor from the hot to the cold end of the furnace, and (3) subsequent nucleation occurred on the walls at the cold end of the ampoule. (Ampoules with and without material sinks at the cold end were investigated.)

Very little information could be found concerning the post-flight results. It was briefly reported that "The number and location of... [the nucleation] sites, as well as the crystal seeds are quantities of interest. The effect of argon gas is to increase the number of nucleation sites and reduce the growth rates." (1, p. 4)

<Note: When Principal Investigator R. Cadoret reviewed this experiment summary in October 1993 and sent back a brief letter highlighting his experiments, he wrote that he had enclosed a publication with the letter which detailed the Spacelab 3 experiment. However, no publication was found in the returned envelope, although Cadoret referred to the publication several times in his letter. Therefore, further details of this experiment are still unclear. Briefly in his letter he mentioned that the experimental conditions in the Spacelab 3 investigation were varied with respect to Spacelab 1 "...but these experiments... [were] also nucleation experiments. No difference was obtained between... [Spacelab 3] and g results, probably because nucleation... [occurred] before reaching the... [stationary] thermal regime...." Without further details of the experiment, this description from the letter was unclear to the editors.>

No further post-flight results could be located at this time, although References (5) and (6) may contain this information. (References (5) and (6) could not be located for review.)

Key Words: Crystal Growth From Vapor, Vapor Transport, Physical Vapor Transport, Vapor Deposition, Binary Systems, Single Crystals, Seed Crystals, Source Material, Thermal Gradient, Vaporization, Sublimation, Condensation, Supersaturation, Mass Transfer, Stefan Flow, Diffusion, Diffusive Mass Transfer, Buoyancy-Driven Convection, Vapor/Solid Interface, Solidification Front Physics, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Nucleation, Nucleation Sites, Gas Pressure, Heat Pipes, Gamma Ray Detectors, Electronic Materials, Semiconductor Applications

Number of Samples: six
Sample Materials: mercury-iodide, HgI_2; host gas: argon (Hg*I*, Ar*)
Container Materials: quartz (each ampoule was approximately 20 cm long and 1.5 cm in diameter)

Experiment/Material Applications:
Please see Cadoret, Spacelab 1.

References/Applicable Publications:

(1) Spacelab 3 Mission Science Review. Proceedings of a Symposium Held at NASA George C. Marshall Space Flight Center, Alabama, Edited by G. H. Fichtl, J. S. Theon, C. K. Hill, and O. H. Vaughan, NASA CP-2429, December 4, 1985, p. 4. (very short summary)

(2) Spacelab Mission 3 Experiment Descriptions. NASA TM-82502, Edited by C. Kelly Hill, November 1982, pp. 9-11. (preflight)

(3) Input received from Experiment Investigator, December 1989 and October 1993.

(4) Omaly, J., Robert, M., Brisson, P., and Cadoret, R.: Mass Transport, Nucleation and Monocrystal Growth of HgI_2, with the Forced Flux Method. Nuclear Instruments and Methods, Vol. 213 (1983), 19-261.

(5) Cadoret, R. and Brisson, P.: The Effect of Gravity on Nucleation, Growth and Physical Transport of HgI_2. CNRS, UA 796, Space Results/CNES-1984.

(6) Brisson, P., Magnan, A., and Cadoret, R.: First Results for HgI_2 Spacelab Experiments SL1 and SL3, Space Results/CNES-1987. (post-flight)

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Co-Investigator(s): GM Vacuum Coating Laboratory (2)
Affiliation(s): (1) Marshall-McShane Designs, Prescott, Arizona; (2) Newport Beach, California

Experiment Origin: USA
Mission: STS Launch #13, STS-017 (STS 41-G, Challenger)
Launch Date/Expt. Date: October 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: NASA Get Away Special (GAS) Canister G-038
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of G-038: Joseph W. McShane, Prescott, Arizona/GM Vacuum Coating Laboratory, Newport Beach, California
Processing Facility: Facility #1: sputter deposition device connected to space vacuum via a vacuum manifold; Facility #2: vapor deposition device connected to space vacuum via a vacuum manifold
Builder of Processing Facility: Unknown, possibly GM Vacuum Coating Laboratory, Newport Beach, California (The glass spheres used in the experiment were blown by the Schott Glass Works Company, Germany.)

Experiment:
Art in Space: Coating of Glass Spheres by Vacuum Deposition Techniques

"For the first time since man first gazed at the stars and from an earth-bound, one atmosphere, one-gravity perspective sought understanding of the unfathomable heavens through his art... [this experiment presented the opportunity] to make use of man's technology as an extension of the artist's eye and hand to venture forth directly into the vacuum and weightlessness of space, seeking understanding." (4, p. 120)

This STS Get Away Special (GAS) experiment "...was created as a unified Arts-Science payload that simultaneously explored the process of vapor deposition in the vacuum and weightlessness of the shuttle environment and created a series of space sculptures utilizing this process, seeking to experience the mysteries of space." (5, p. 267)

Three separate investigations were performed in the canister. Two of the investigations employed vacuum deposition techniques (vapor deposition and sputter deposition) and are reported in this experiment summary; the third investigation is described under McShane, STS-017 "Art in Space: Sampling and Artistic Preservation of the Space Vacuum" (Chapter 18).

During vacuum thin-film metallic deposition, a vaporized metallic element coats whatever substrate is in its path. If most of the molecules in the vapor reach the substrate without first striking another molecule, the resultant coating is reasonably dense and adherent.

Reportedly, it was expected that "By working at short distances in a clean vacuum of less than $1X10^{-5}$ torr, about 1/10,000,000 of an atmosphere... [excellent coatings would be produced] in spite of some restrictions in the pumping speed caused by the restricted openings in the NASA [Gas Canister] lid and thermal cover." (4, p. 123) In the space environment "...We can pump for several days without using power or adding contamination from organic oils often used in conventional vacuum pumps." (4, p. 123) Further, materials can be evaporated from filaments which (in a one-gravity environment) tend to drip from the source during the heating process and fall onto the substrate.

(1) Vapor Deposition

The objective of the first investigation was to use a vapor deposition process to create three spherical sculptures interiorly coated with metallic films. Three, 3000-ml glass spheres were selected. Prior to the shuttle flight, each of the spheres was configured with a tungsten filament which had been coated or wrapped with metal.

It appears that during the space experiment, the interior of each sphere was exposed to the space vacuum and the tungsten filament was heated. Subsequently, the metal vaporized and the vapor covered the interior of the glass spheres.

Post-flight analysis of the equipment dedicated to this investigation indicated that the vapor deposition experiments were performed as planned "...creating two sculptures with very clean opaque coatings of gold and aluminum and one sculpture with a coating deposited from a two stage filament coated with aluminum and silicon monoxide, creating an iredescent [sic] effect." (5, p. 269)

<Note: No detailed results of this experiment were presented which compared the quality of the space-produced films with Earth-produced films. A very short mention of the space-produced films in Reference (7) reported that "...[the] deposition process was similar to that used on Earth to coat lenses, glass, and mirrors, but the vacuum and weightlessness of space allowed a highly uniform coating that was just a few microns thick." (7, p. 23)>

<Note: It was not clear if the films mentioned in Reference (7) referred only to the vapor deposition films, the sputter deposited films (see next section), or both types of films.

Further, although it was not specifically stated in the available references, it appears that the first two spheres were each coated with one metallic element (one sphere was coated with gold, the second with aluminum).>

(2) Sputter Deposition

The objective of the second investigation was to use a sputter deposition process to create five spherical sculptures interiorly coated with metallic films of gold, silver, platinum, and chrome. To achieve this goal, the interior of five, 500-ml glass spheres were each configured with the appropriate sputter deposition hardware. (Reportedly, the experiments were performed in smaller spheres than employed for the vapor deposition experiments because during sputtering, the substrate is usually much closer to the source.)

During the experiment, the interior of each sphere was exposed to the space vacuum environment. Then, "By accelerating positively charged argon ions into the surface of a negatively charged metal target mounted in the center of each sphere, target molecules were [mechanically knocked loose from the relatively cold source (the target) and] ejected by the impact of the argon ions and formed a coating on the inside of each sphere over a period of hours." (5, p. 268)

<Note: Although it was not specifically stated in the available references, it appears that each sphere was coated with a single metallic element. One sphere was coated with gold, a second with silver, a third with platinum, and a fourth sphere with chrome. The fifth sphere was probably coated with one of these metallic elements.>

Post-flight analysis of the equipment dedicated to this investigation indicated that the experiment "...functioned in the expected manner but did not create coatings as dense as anticipated. [The reason for this anomaly]... was traced to apparent arching in the vacuum manifold during the ionization process, which limited the charge being transmitted to the target. Thus, sculptures that were intended to have metal coatings applied in space ranging from opaque to semitransparent, returned with all semitransparent coatings instead; an unexpected but not unpleasing result." (5, p. 268)

No detailed results of the sputter deposition experiment were presented which compared the quality of the space-produced films with Earth-produced films.

Key Words: Crystal Growth From Vapor, Vacuum Film Deposition, Vapor Deposition, Vaporization, Sublimation, Sputter Deposition, Vapor/Solid Interface, Coated Surfaces, Spheres, Films, Thin Films, Metals, Art, Source Material, Substrates, Direct Exposure to Space Environment, Vacuum, Space Vacuum, Hardware Malfunction

Number of Samples: Vapor deposition: three; sputter deposition: five
Sample Materials: Vapor deposition: It appears one of the spheres contained a filament coated with gold, another contained a filament coated with aluminum, and that the remaining contained a filament coated with aluminum and silicon monoxide. Sputter deposition: films created from gold, silver, platinum, and chrome (see note in the above experiment summary).
(Au*, Al*, Al*Si*O*, Ag*, Pt*, Cr*)
Container Materials: glass spheres

Experiment/Material Applications:
"The methods and results of G-38 supply useful data for simplified coatings of large antennae, heat shields, solar collectors and optical mirrors in space; where size is not limited to the confines of a vacuum chamber." (5, p. 267)

Advantages of using the available space vacuum are detailed in the above experiment summary.

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (preflight; very short description)

(2) Space Shuttle Mission 41-G. NASA Press Kit, October 1984, pp. 23-24. (preflight; very short description)

(3) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, September 4, 1984. (short description; preflight)

(4) McShane, J. W. and Coursan, C. D.: An Artist's Exploration of Space. In NASA Goddard Space Flight Center's 1984 Get Away Special Symposium, August 1-2, 1984, NASA CP-2324, pp. 119-126. (preflight)

(5) McShane, J. W.: Art in Space -- A Divergent Exploration. In Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, NASA CP-2401, pp. 267-273. (post-flight)

(6) Shuttle Payload Creates Space Sculpture, AW&T, October 15, 1984.

(7) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team, 1989, p. 23. (post-flight; very brief description)

(8) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

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Experiment Origin: USA
Mission: STS Launch #17, STS-024 (STS 51-B, Spacelab 3: Challenger)
Launch Date/Expt Date: April 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack
Processing Facility: Vapor Crystal Growth System (VCGS): "The Furnace/Ampoule Unit provides housing for the crystal/source material and provides a spatially-unique temperature distribution on the ampoule." (8, p. 7-3)
Builder of Processing Facility: TRW, Redondo Beach, California

Experiment:
Mercuric Iodide Vapor Crystal Growth System (VCGS)

The performance of single crystalline sensing elements in detector devices (e.g., x-ray and gamma ray detecting instruments) is determined by the density of structural defects in the material. Some of the sources of these defects are related to (1) irregular gravity-driven convection during the vapor transport process and (2) gravity-induced mechanical crystal slippage.

Under normal gravity conditions, the transport of the vapor to the growing crystal during vapor crystal growth is achieved by diffusion and convection. As long as these mechanisms are constant with time, a crystal of good quality is expected. However, when the convective transport becomes irregular with time, the crystal surface is exposed to varying supersaturations of the vapor and crystal irregularities result.

Mechanical slippage occurs when the weight of the extremely fragile crystal causes a "cold flowing" or slippage of the material (during or after processing). This gravity-induced phenomenon results in a further increase in the density of defects.

It was proposed that growth of HgI_2 crystals under low-gravity conditions would result in large, single crystals which have a substantial reduction in defect density over terrestrial-grown crystals.

This Spacelab 3 experiment was designed to investigate the vapor crystal growth of mercuric iodide (HgI_2) under low-gravity conditions. The specific objective of the investigation was to produce an HgI_2 crystal of high quality by a technique of vaporization and recondensation.

The low-gravity experiment was performed in the Spacelab 3 Vapor Crystal Growth System (VCGS). The center of the VCGS furnace was configured with a cylindrical ampoule. A pedestal was attached to one end of this ampoule to accommodate a small terrestrial-grown seed crystal (3 mm per side). The pedestal fit over a metal support tube which contained a Peltier cooling device. The source material (polycrystalline HgI_2) was located at the sides of the ampoule, and was held in place by glass fingers. (The source material had been prepared prior to launch by a "...fairly extensive series of purification steps. These included multiple sublimation/condensation steps and a melting/refreezing operation." (Reference 11))

The thermal profile of the ampoule was achieved by two independently controlled heating coils. A circular coil covered with a heat equalization ring (configured in the payload below the ampoule at the pedestal end) heated the lower section of the ampoule. A helically wound vertical coil provided thermal equalization along the vertical walls of the ampoule. On top of the helical coil was a reflective shield for minimizing heat loss. This arrangement resulted in a temperature minimum at approximately the middle of the ampoule.

The entire apparatus was enclosed by a bell jar of high optical quality, which allowed microscopic observation of the growing crystal. (See References (6), (8), and/or (9) for further details of the hardware.)

During the mission, the VCGS was used to produce a single crystal of mercuric iodide. The experiment procedure consisted of three basic steps: (1) heat-up (4 hours), (2) growth (118 hours), and (3) cool down (8 hours).

During the heat-up phase, the temperature at the source, heat distribution ring, and cooling sting were controlled to optimize the growth conditions. Gradually decreasing the pedestal temperature initiated the growth phase. (The temperature difference between the source material and crystal was in the region of a few degrees celsius (<10 degrees).) Periodic etching of the growing crystal was possible by oscillating the temperature of the source material. This etching was performed to remove any stray particles of mercuric iodide dust on the seed's surfaces. (The technique reportedly also allowed more stable crystal growth and avoided spurious crystal nucleation.) System cool-down was in-

itiated when the experiment time had elapsed (Spacelab shutdown).

"Notwithstanding the efforts made to predetermine the proper growth conditions on the ground (elimination of convection around the ampoule by evacuation of the space between the ampoule and bell jar), the temperature again had to be adjusted to obtain crystal growth in space. This accounts for the fact that during the first 14 hours in space no growth was observed. The temperature difference between the source and the pedestal had to be increased by 1.6 ^{o}C to initiate growth.

"As growth continued, it became clear that much higher temperature gradients could be applied than is usually done on the ground, without disturbing the stability of the crystal surfaces or without causing any new nucleation. Using the higher temperature gradients it was therefore possible to obtain higher growth rates by diffusion transport alone than is usually seen on the ground with both diffusion and convection transport...." (9, p. 278)

"...the linear growth rate of the of the crystal varied, reaching a maximum of about 3 mm/day. This compares to growth rates on earth that range from about 0.5 to 2 mm/day, with an average of about 1 mm/day. (On earth it is difficult to achieve a rate of 3 mm/day without rapid occurrence of undesired spurious nucleation)." (7, p. 14)

Post-flight examination of the 7.2 gram, 1.2 cm X 1.2 cm X 0.8 cm space-grown HgI_2 crystal was performed using gamma-ray rocking curve analysis and electronic measurements. The rocking curve measurements revealed a single peak indicative of a single crystal without major grain boundaries. However, evidence of internal strain, as determined by the width and asymmetrical shape of the curve, was also detected. The rocking curve of an Earth-grown crystal revealed several peaks indicating that the crystal consisted of three sections at slight angles with respect to each other (see Reference (9) for rocking curves).

The crystal quality of radiation detectors can be determined by examining the electron and hole mobilities and lifetimes. These properties were investigated by evaluation of the nuclear spectral response as a function of applied bias. Comparison of these values to those for detectors produced on Earth revealed improved properties for the low-gravity material (see Reference (9)). This was especially true "...with respect to the holes, which is the most critical factor for detector operation." (9, p. 282)

"Initial electrical properties measurements on slices of the space-grown crystal indicated extremely high electron and hole mobilities, more than twice as high as the best ever reported on earth. When these measurements were repeated some 3 months later, the mobilities had degraded to typical earth-grown values. This is not understood although since this particular material is extremely soft and easily damaged, the apparent degradation could have been caused by aging and handling despite the extreme precautions taken to prevent such effects." (3, p. 29)

It was concluded that the experiment to grow HgI_2 from the vapor phase performed exceptionally well, and the objectives were fulfilled. Growth conditions in space allowed processing with an increased temperature gradient, such that the growth rate with diffusion transport alone was larger than on Earth (diffusion and convection). The structural quality and electronic transport properties of the low-gravity sample were significantly improved over the 1-g processed material.

Other interesting results are reported in several of the available publications.

Key Words: Crystal Growth From Vapor, Vapor Transport, Vapor Deposition, Binary Systems, Single Crystals, Seed Crystals, Source Material, Thermal Gradient, Vaporization, Sublimation, Condensation, Supersaturation, Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Thermal Oscillations, Vapor/Solid Interface, Growth Rate, Crystalline Structure, Crystal Morphology, Surface Morphology, Grain Boundaries, Crystalline Strain, Stress, Mechanical Slippage, Sample Purity, Defect Density, Nucleation, Spurious Nucleation, X-Ray Detectors, Gamma Ray Detectors, Electrical Properties, Electron Mobility, Electronic Materials, Deterioration of Samples After Low-G Flight

Number of Samples: one
Sample Materials: mercury-iodide, HgI_2 (Hg*I*)
Container Materials: Ampoule and its pedestal region: borosilicate type glass (e.g., Pyrex™)

Experiment/Material Applications:
"Mercuric Iodide is a material used for the fabrication of the sensing element in solid state x-ray and gamma ray detecting instruments. The advantage of the use of mercuric iodide in such instrumentation is such that the systems are able to operate at room temperature, compared with systems based on germanium and silicon which need cooling to liquid nitrogen temperature." (9, p. 270)

References/Applicable Publications:
(1) van den Berg, L., Schnepple, W. F., Skinner, N., and Ortale, O.: Crystal Growth Experiments on SL3. In The Sixth American Conference On Crystal Growth, Atlantic City, New Jersey, July 15-20, 1984. (preflight)

(2) Spacelab 3 Early Results. Spaceflight, Vol. 27, 1985, p. 361. (post-flight)

(3) Naumann, R. J.: Microgravity Science and Applications Program in the United States. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUC), pp. 28-29. (post-flight)

(4) Schnepple, W. F., van den Berg, L., and Skinner, N.: Growth of HgI2 Single Crystals in Spacelab III. Journal of Spacecraft, Vol. 16, November-December 1979, pp. 441-443. (preflight)

(5) van den Berg, L. and Schnepple, W. F.: Growth of Mercuric Iodide in Spacelab III. 19th International SAMPE Technical Conference, October 13-15, 1987. (post-flight)

(6) Materials Processing in Spacelab 3. Application Payload Projects, Spacelab Payload Project Office, Marshall Space Flight Center, Huntsville, Alabama. (preflight)

(7) Schnepple, W., van den Berg, L., Skinner, N., and Ortale, C.: Spacelab 3 Vapor Crystal Growth Experiment. In Spacelab 3 Mission Science Review, proceedings of a symposium held at NASA George C. Marshall Space Flight Center, Alabama, December 4, 1985, NASA CP-2429, pp. 12-17. (see also short summary, p. 4) (post-flight)

(8) Vapor Crystal Growth System. In Catalog of Selected Spacelab Experiment and Support Equipment, Vol. 1, developed for NASA/Office of Space Sciences and Applications, November 1989, Teledyne Brown Engineering, Huntsville, Alabama, pp. 7-2 - 7-3. (processing facility)

(9) van den Berg, L. and Schnepple, W. F.: Growth of Mercuric Iodide (HgI_2) for Nuclear Radiation Detectors. In Microgravity Science and Applications Flight Programs, January-March 1987, Selected Papers, NASA TM-4069, Vol. 1, pp. 269-287. (post-flight)

(10) Schnepple, W. F., van den Berg, L., and Skinner, N.: Growth of HgI_2 Single Crystals in Spacelab III. 17th AIAA Aerospace Sciences Meeting, New Orleans, Louisiana, January 15-17, 1979, AIAA Paper #79-0307, 4 pp. (preflight)

(11) Input received from Principal Investigator W. F. Schnepple, August 1993.

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Co-Investigator(s): Cook, E. (2)
Affiliation(s): (1,2) 3M Corporation, St. Paul, Minnesota

Experiment Origin: USA
Mission: STS Launch #20, STS-027 (STS 51-I, Discovery)
Launch Date/Expt. Date: August 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
The payload was housed in a NASA Experimental Apparatus Container (EAC), mounted on the aft bulkhead in the middeck.
Processing Facility: Physical Vapor Transport of Organic Solids (PVTOS) Facility: nine independent experimental cells, each housing a Pyrex™/stainless steel ampoule. Each ampoule contained an organic source material at the hot end and a temperature-controlled substrate at the cooler end.
Builder of Processing Facility: 3M Corporation, St. Paul, Minnesota

Experiment:
Physical Vapor Transport of Organic Solids (PVTOS-1)

During physical vapor transport (PVT) crystal growth, a material is (1) heated to sublimation at the hot end of the furnace, (2) diffuses through a buffer gas across which is imposed a temperature gradient, and (3) condenses at the cold end of the furnace. On Earth, both buoyancy-driven convection and gaseous diffusion of the vapor contribute to the formation of the crystalline product. In a reduced-gravity environment, the convective contribution should be greatly suppressed, resulting in vapor growth which is strongly driven by the diffusion contribution. Such diffusion-controlled growth contributes to the production of higher quality crystals.

This experiment was the first in a series of investigations designed by Debe et al. to study the closed-cell vapor transport of organic solids. The specific objectives of the experiment were to (1) study the vapor transport process, (2) explore the effects of low-gravity processing on film microstructure, and (3) evaluate the thermal performance of the experimental hardware under space flight conditions.

A total of nine, sealed, ultra high vacuum (UHV)-quality stainless steel PVT ampoules were employed during the mission. Five of these ampoules were dedicated to the study of the vapor transport process and four to the growth of thin films of model heterocyclic organic compounds on specialized substrates. (See the **Sample Materials** section (below) for ampoule contents.)

During the experiment, the ampoules were heated to 400 ^{o}C by concentric heater assemblies. The heater power, source end, cell surface, and substrate temperatures, were controlled and monitored during 4.5-hour processing periods. It was noted that "The outgassing of the cell's contents and resultant heat loss by gas phase conduction... [were] important parameters for the cell power requirements and ampoule boundary conditions." (9, p. 2406 (abstract))

Post-flight, space-based experimental results were compared to ground-based control experiments which (1) were conducted in "hot end up" and "hot end down" orientations and (2) duplicated the flight experimental conditions.

Reportedly, "Very well defined substrate deposits, both thick and thin film, were obtained in all nine of the PVTOS flight PVT ampoules, demonstrating a clear dependence on the molecular weight of the buffer gas." (9, p. 2406) Extensive characterization of the flight-produced films by IR and UV-visible reflection-absorption spectroscopy, X-ray diffraction, interferometry, ellipsometry, X-ray photoemission, SEM and visible microscopy (1) established the existence of a new polymorph of copper phthalocyanine and (2) indicated that low-g processing results in films with enhanced uniaxial ordering, smoothness, and optical homogeneity.

"An assessment of the [flight] PVTOS experimental cell design and performance indicates the use of UHV-type materials and sealing techniques, including a glass-to-metal joint, have very adequately met the launch and landing stresses while providing the desired thermal insulation and environment for a PVT experiment with limited power available. The outgassing of materials in our particular cell design was also typical of UHV systems, with H_2 the dominant species, confirming our cell cleaning and assembly procedures. Heat transfer from the cell heater to the outer stainless-steel envelope by gas phase conduction in microgravity is significant when the residual pressures exceed 0.5 Torr. (9, p. 2410)

<Note: Not all of the references listed below were available to aid in the preparation of this experiment summary.>

Key Words: Crystal Growth From Vapor, Vapor Transport, Physical Vapor Transport, Vapor Deposition, Films, Thin Films, Film Growth, Film Microstructure, Organic Systems, Organic Crystals, Ordered Organics, Model Materials, Source Material, Substrates, Thermal Gradient, Vaporization, Sublimation, Condensation, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Vapor/Solid Interface, Sample Microstructure, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Gas Pressure, Outgassing, New Polymorphs, Optical Characterization, Vacuum

Number of Samples: nine ampoules
Sample Materials: Organic materials
Source Materials: (a) copper phthalocyanine; (b) N, N-bis(3,5-xylyl)perylene-3, 4:9, 10-bis(dicarboximide); (c) N, N-bis(2-phenethyl)perylene-3, 4:9, 10-bis(dicarboximide)
Buffer Gases: Helium and Xenon
Substrate Materials: (1) Bare copper or (2) oriented thin films of metal-free phthalocyanine
(He*, Xe*, Cu*)
Container Materials: Pyrex^{TM}/stainless steel ampoules
<Note: It is not clear which sections of the ampoules were comprised of Pyrex^{TM}.>

Experiment/Material Applications:
This research was designed to investigate the effect of convection (in a low pressure gas transport system) on the microscopic physical structure of thin films and crystals grown from the vapor phase.

Reportedly, the new films would be used for "Basic research, but the phthalocyanines have important uses in optical data storage, to name one application." (11)

References/Applicable Publications:
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(2) Debe, M. K., Poirier, R. J., Cook, E. L., Miller, L. R., Spiering, M. S., and Floeder, S. P.: Gas Phase Evolution and Convection in the Closed Cell Vapor Transport Growth of Organic Thin Films in Microgravity and Unit Gravity. J. Vac. Sci & Technol., Vol. A8(1) (1990), 49. (post-flight)

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(14) Debe, M. K.: Low Gravity Enhanced Growth of Phthalocyanine Polymorph Films, U.S. Patent No. 5,071,691.

<Note: Additional publication titles are available from M. K. Debe.>

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Principal Investigator(s): Debe, M. (1)
Co-Investigator(s): Cook, E. (2)
Affiliation(s): (1,2) 3M Corporation, St. Paul, Minnesota

Experiment Origin: USA
Mission: STS Launch #26, STS-26, (Discovery)
Launch Date/Expt. Date: September 1988
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Middeck Experiment
The payload was housed in a NASA Experimental Apparatus Container (EAC), mounted on the aft bulkhead in the middeck.
Processing Facility: Physical Vapor Transport of Organic Solids (PVTOS) Facility-nine independent experimental cells, each housing a Pyrex™/stainless steel ampoule. Each ampoule contained an organic source material at the hot end and a temperature-controlled substrate at the cooler end.
Builder of Processing Facility: 3M Corporation, St. Paul, Minnesota

Experiment:
Physical Vapor Transport of Organic Solids (PVTOS-2)

This experiment was the second in a series of investigations designed by Debe et al. to study closed-cell vapor transport of organic solids (see Debe, STS-027). The specific objective of the investigation was to produce organic thin films with ordered crystalline structures using the physical vapor transport (PVT) crystallization technique.

The Principal Investigator reported that (1) a detailed post-flight description of the experimental setup is provided in Reference (3) and that (2) subsequent space results have not yet been published, but have been presented orally. <Note: Reference (3) was not available at the time this experiment summary was finalized for publication.>

A document written prior to the flight (Reference (1)) indicated that the space experiments were to be performed within nine independent PVT ampoules. During the flight, an organic material in each ampoule was to be vaporized. The vapor was then expected to migrate through a buffer gas and recondense on a flat surface at the cooler end of the ampoule. (Expected processing temperatures, ampoule contents, etc. were not cited in the preflight reference.)

Post-flight analysis of the resultant crystalline films were to include an examination of (1) ordered structure, (2) optical characteristics, and (3) microstructure.

The Principal Investigator reported that the results of the STS-26 experiment "Fully substantiated and added to the results of the PVTOS-1 experiments. The physical vapor transport deposited films in microgravity were more highly ordered than the ground controls, consisted of purer polymorphic forms, and consisted primarily of the new copper phthalocyanine crystal form called M-CuPc. Many interesting effects due to the varying buffer gas molecular weights and thermophysical gas properties were observed. Publication of all these results is still intended." (4)

No further information concerning the results of this experiment appear to be available at this time.

Key Words: Crystal Growth From Vapor, Vapor Transport, Physical Vapor Transport, Vapor Deposition, Films, Thin Films, Film Growth, Film Microstructure, Organic Systems, Organic Crystals, Seed Crystals, Ordered Organics, Source Material, Substrates, Thermal Gradient, Vaporization, Sublimation, Condensation, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Vapor/Solid Interface, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Gas Pressure, New Polymorphs, Optical Characterization

Number of Samples: nine ampoules
Sample Materials: Organic materials, buffer gas, specialized substrates, specifically:
Source Materials: Copper phthalocyanine (all nine ampoules)
Buffer Gases: He, N_2, CF_4, Xe (at various pressures)
Substrates: Predeposited "seed" films of metal-free phthalocyanine, oriented in two orthogonal crystallographic orientations.
Container Materials: Pyrex^{TM}/stainless steel ampoules

Experiment/Material Applications:
See Debe, STS-027

References/Applicable Publications:
(1) Space Shuttle Mission STS-26. NASA Press Kit, September 1988, p. 19. (preflight)

(2) "Seven Marshall Payloads to Fly on STS-26 in June." In Marshall Star, Vol. 28, No. 5, October 7, 1987, pp. 1-2. (preflight; very short description)

(3) Debe, M. K., Poirier, R. J., Schroder, F. S., Cook, E. L., and Follett, G. J.: Design and Performance of a Vapor Transport Cell for Operation Onboard the Space Shuttle Orbiter. Rev. Sci. Instrum. Vol. 61(2) (1990), p. 865. (experimental setup)

(4) Input received from Principal Investigator M. K. Debe, August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: STS launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, M E D E A Double Rack
Processing Facility: Monoellipsoidal Mirror Furnace (ELLI)
Builder of Processing Facility: Dornier Systems, Friedrichshafen, Germany

Experiment:
Vapor Zone Crystallization of Cadmium Telluride (MD-ELI 03)

During ground-based vapor crystal growth, gravity-induced thermal and solutal convective flows in the zone between the feed and growing crystals result in oscillatory and turbulent flows at the crystalline interface. These flows, in turn, often contribute to the formation of microinhomogeneities in the processed materials. Such inhomogeneities tend to degrade crystalline quality. In contrast, during space-based vapor crystal growth, gravity-induced convective flows are reduced and diffusion-dominated flow of the vapor components (Cd atoms, Te_2 molecules) should result.

The objectives of this Spacelab D1 experiment were to (1) produce a CdTe crystal grown from the vapor phase in a low-gravity environment, and (2) determine (a) if diffusion-dominated vapor crystal growth had occurred and (b) if crystals with a reduced defect density had been produced.

"The basic [experimental] set-up consisted of a quartz ampoule... containing a cylindrical CdTe seed and a feed rod, separated by a distance of 6mm. After equilibration of the two phase boundaries with the vapor zone in the focus of the monoellipsoidal mirror furnace "ELLI," sublimation growth was to be initiated by translation of the ampoule at a rate of 0.35mm/h." (4, p. 288)

Reportedly, "Due to initial difficulties in activating the ELLI furnace, the experiment could not be performed as scheduled." (1, p. 62) However, the experiment was finally performed at the end of the mission, but was subjected to several restrictions/alterations including a reduction of growth time, a reduction in lamp power and a change of furnace atmosphere from 0.1 bar argon to 1 bar air.

During the mission, "A 2.4 mm thick monocrystalline layer of CdTe was grown from the vapor phase by sublimation onto a cylindrical seed of 15mm diameter within 9.6 hours. Despite... [the] poor quality of the seed, a remarkable decrease in the etchpit density was noted. However, the values reached were not smaller than those obtainable on earth from seeds of high quality. Due to the small growth length, it cannot be said whether a further improvement would have... [occurred] under... [low-gravity]." (4, abstract, p. 288) <Note: The reasons why the flight seed crystal was poor were not clear.>

No further information concerning this experiment could be located at this time.

Key Words: Crystal Growth From Vapor, Vapor Transport, Vapor Deposition, Films, Thin Films, Film Growth, Binary Systems, Feed Material, Seed Crystals, Vaporization, Sublimation, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Thermosolutal Convection, Buoyancy-Driven Convection, Turbulent Flow, Vapor/Solid Interface, Translation Rate, Crystalline Structure, Crystal Homogeneity, Crystal Morphology, Surface Morphology, Defect Density, Etch Pits, Electronic Materials, Semiconductor Applications, Semiconductors, Gamma Ray Detectors, Infrared Detector Applications, Incomplete Sample Processing, Furnace Malfunction

Number of Samples: one
Sample Materials: cadmium-telluride
(Cd*Te*)
Container Materials: quartz ampoule
(Si*O*)

Experiment/Material Applications:
The semiconductor CdTe has applications as an "...infrared window, electrooptic modulator, photovoltaic material and in particular as a sensitive... [gamma] ray detector and spectrometer." (2, p. 121)

References/Applicable Publications:

(1) Bruder, M., Dian, R., and Nitsche, R.: Vapour Zone Crystallization of Cadmium Telluride (Expt: D1-MD-ELLI 03). In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 62. (abstract only)

(2) Bruder, M. and Nitsche, R.: Vapour Zone Crystallization of Cadmium Telluride. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 121-122. (preflight)

(3) Bruder, M. and Nitsche, R.: Seeded Vapour Growth of Cadmium Telluride Using Focused Radiation Heating. Journal of Crystal Growth, Vol. 72, 1985, pp. 705-710. (related research)

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(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, p. 48. (post-flight; acceleration measurements on D1)

(6) Input received from R. Schwarz (Kristallographisches Institut), July 1993.

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Co-Investigator(s): Unknown
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Experiment Origin: France
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Gradient Heating Facility (GHF)
Builder of Processing Facility: CNES (Centre National d'ETudes Spatiales), Toulouse, France

Experiment:
Solutal and Thermal Convection in Ge-I2 Vapor Phase-(WL-GHF 05 and WL-GHF 06)

The Chemical Vapor Tranpsort (CVT) crystal growth technique is widely used to grow high quality crystals of electronic materials at low temperatures (e.g., Si, GaAs, HgI_2). In order to increase the quality of the grown crystals, the governing parameters of the process must be known. These parameters include both gravity-dependent (convective flows) and quasi-diffusive transport mechanisms.

The objectives of this Spacelab D1 research were to (1) investigate and characterize the parameters which govern the vapor phase transport during vapor crystal growth and (2) employ these parameters in theoretical analyses.

Each sample consisted of (1) a growth ampoule, (2) a source, (3) a sink, (4) a substrate, and (5) a transport agent. The growth ampoules were made of silica glass (19.5 mm outside diameter). The source and sink were Ge polycrystals held at the tube ends by a Ge grid. The Ge monocrystalline substrate was held by a quartz holder sealed on the wall of the ampoule. GeI_4 was used as the transport agent. Six chromel-alumel thermocouples, located at the quartz wall, were used for temperature field determination.

During the mission, the Spacelab Gradient Heating Facility (GHF) was used to process two samples based on a "three-zone" arrangement (source, deposit, and sink). This configuration allowed the "...release [of] the hydrodynamic regime from thermodynamic conditions through the creation of a diffusional forced flow between the source and the sink at different temperatures." (4, p. 323) The sample materials were also processed on the ground for com-

parison.

Post-flight examination of the thermal flight data indicated "An excessive temperature of the middle heating zone... attributed to the superisolating (power consumed is zero)[,] led to the setting up of practically linear thermal gradients between the source and the sink...." (4, p. 325) <Note: The exact meaning of this quote was unclear to the editors.> It was also reported that at the source, there was little change in the mass flux value with tetraiodide mass. "Quantities deposited on the substrate are small and the substrate present sometimes a slightly attack [sic]." (4, p. 325) <Note: the meaning of this quote is unclear to the editors.> It was indicated that the substrate temperature was too high, which resulted in a reduction in the supersaturation and, consequently, a restiction in the epitaxy. The small quantity of deposited material was attributed to the thermal behavior of the GHF.

Despite the detrimental thermal behavior of the GHF, the experiments illustrated that (1) the quality of the substrate processed in space was better than that processed on Earth and (2) a purely diffusive regime was present in the low-gravity experiments (determined via examination of the mass flux rates).

The space results were used to obtain kinetic parameters of the transport reaction via a one-dimensional theoretical model, which accounted for non-equilibrium interfacial transfer. <Note: Reference (4) contains the model description and results.> Reportedly, the transport velocity approaches thermodynamic equilibrium (i.e., the Peclet number approaches infinity) as the mass of the transport agent increases. "This is most probably due to the fact that the pressure being proportional to the mass of the tetraiodide, at high pressure the probability of occupation of a vacant site [located on the substrate] is higher, the surface coverage of the growth sites is greater and the limiting action of the surface kinetics decreases." (4, p. 330)

It was reported that future modeling would use the two-dimensional Navier-Stokes equations. However, no publication concerning this work could be located.

Key Words: Crystal Growth From Vapor, Vapor Transport, Chemical Vapor Transport, Vapor Deposition, Epitactic Layers, Binary Systems, Single Crystals, Source Material, Transport Agent, Substrates, Thin Films, Thermal Gradient, Vaporization, Supersaturation, Sublimation, Condensation, Mass Transfer, Heat and Mass Transfer, Diffusion, Diffusive Mass Transfer, Thermal Diffusion, Thermal Equilibrium, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Thermosolutal Convection, Vapor/Solid Interface, Crystalline Structure, Crystal Morphology, Surface Morphology, Gas Pressure, Electronic Materials, Semiconductor Applications, Semiconductors, Furnace Malfunction, Thermal Environment More Extreme Than Predicted

Number of Samples: two
Sample Materials: Ge-GeI_4 (iodides used as transporting agents); source and sink: polycrystals of Ge; substrate: monocrystal of Ge (Ge*I*)
Container Materials: silica glass
(Si*O*)

Experiment/Material Applications:
It was reported that "...the epitaxy of Ge in the GeI_2/GeI_4 system... appears to be simple and a good candidate to study growth mechanisms." (4, p. 323)

References/Applicable Publications:
(1) Launay, J. C. and Debegnac, H.: Ge/GeI4 Chemical Growth in Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 47-59. (post-flight; abstract only)

(2) Launay, J. C. and Debegnac, H.: Ge/GeI4 Chemical Growth in Microgravity. Solutal and Thermal Convection in Ge-I2 Vapour Phase. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 122-124. (preflight)

(3) Cambdon, G., Castets, B., Boutemy, O., Giequel, J. P., Aubron, R., Boddnert, M., and Benoit, J.: Gradient Heating Facility Behavior. In ESA 5th European Symposium on Material Science Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 115-119. (post Spacelab 1, prior to D1)

(4) Launay, J. C., Debegnac, H., Zappoli, B., and Mignon, C.: Germanium Epitaxial Growth in Closed Ampoules. Journal of Crystal Growth, Vol. 92, 1988, pp. 323-331. (post-flight)

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Co-Investigator(s): Thompson, E. D. (Customer) (3), Flocton, I. (Payload Manager) (4), Hoffer, D. (5)
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Experiment Origin: Canada
Mission: STS Launch #23, STS-031 (STS 61-B, Atlantis)
Launch Date/Expt Date: November 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: High School Student Experiment
Get Away Special (GAS) canister G-479
Volume of Canister: 2.5 cubic feet
Location of Canister: STS Payload Bay
Primary Developer/Sponsor of this experiment within G-479: Canadian High School Students/Telesat Canada, Ontario, Canada
Processing Facility: Six vapor deposition tubes
Builder of Processing Facility: Telesat Engineers, Ottawa, Ontario, Canada

Experiment:
Primary Mirror Production Using Vapor Deposition on a Quartz Plate

During the processing of mirrors on Earth, oxidation of the employed metals lowers the quality of the reflecting surface. If the mirrors could be processed in the contamination-free environment of space, higher quality mirrors might result.

This experiment was one of two investigations housed within the G-479 Get Away Special canister during STS-031. The other experiment (of the two) is also applicable to this data base (see Lipsett, STS-031 (Chapter 9)).

The specific objectives of the research were to (1) create mirrors in a low-gravity environment and (2) compare the optical properties of the space-produced mirrors with terrestrial-produced mirrors.

The experimental setup was configured to manufacture the mirrors using a technique widely used in the optics industry. The technique employs a tungsten filament which has been wetted with an element (such as gold or silver), an evacuated chamber, and a substrate. During the process, (1) the filament is powered, heating the element, (2) the element vaporizes, and (3) the vapor radiates in all directions coating everything (including the substrate) with a thin film. After sufficient accumulation of

the vapor is realized on the substrate, a reflective surface is formed.

On Earth, high quality mirrors are generally produced in vacuums of the order of 1 to 5 micro-Torr. In contrast, the vacuum available in the shuttle's "parking orbit" was expected to be of the order of 2 to 5 milli-Torr. Nevertheless, the investigators decided to use the vacuum conditions available in the shuttle's orbit (for all but one of the experiments). This decision allowed them to investigate the quality of the processing available in the shuttle "parking orbit" environment.

Prior to the shuttle launch, six tubes were configured for the experiments. Five of the tubes were used to sublimate metallic elements; the sixth tube was used to sublimate sodium.

The five tubes dedicated to the metallic elements were made of steel. Each tube contained (1) two, 2-inch diameter quartz lenses (one at each end acting as the substrates), (2) a coated filament (inserted between the lenses), and (3) "inserts holding smaller samples... [which] were later examined for their metallurgical properties by performing transmission electron microscopy (TEM) tests." (4, p. 53) Gold, silver, and aluminum were chosen as filament coatings. Holes were drilled into the tube that "...allowed air to evacuate from the tube when the shuttle left the earth's atmosphere (the GAS cannister [sic] was open to the shuttle bay)." (4, p. 52) It was noted that there was a drawback in selecting this evacuation design because air would re-enter the tubes when the shuttle returned to Earth (and would thus permit oxidation and contamination of the samples).

Because sodium requires a vacuum on the order of 1 to 6 micro-Torr to sublimate, the remaining (sixth) sealed tube was evacuated prior to launch. The tube was made of glass and was designed with an hour-glass shape. The neck of the hour-glass held the sodium to be vaporized; the filament was wrapped around the neck. The two ends of the hour-glass acted as the substrate for the desired mirrors. "The packaging for the hourglass tube was designed to absorb the vibration of launch and contain the chemically reactive sodium if the glass were to break.... Two drawbacks to this hourglass tube were the fragility of the glass and the fact that helium permeated the glass walls degrading the vacuum over time. This made it imperative that the flight tube be manufactured and installed as late as possible in the experiment's integration into the shuttle." (4, p. 52)

Post-flight analysis of the payload indicated that metallic mirrors were made in all.of the steel tubes. Unfortunately, sodium mirrors were not made. It was determined that the neck of the hour-glass tube cracked during launch and the required vacuum to

sublimate the sodium was lost.

After the space experiment was returned to Earth, similar mirror production experiments were performed in the space hardware. These experiments were performed in a thermal vacuum chamber.

At the time Reference (4) was published, full evaluation of the mirror samples had not been completed. Tests were still being performed on the Earth and space-processed samples in the research labs of the National Research Council of Canada, the University of Toronto, and Litton Systems (Toronto). It was reported that the tests were to include: "...reflectance measurements, polarization measurements, backscatter measurements, diffraction tests, transmission electron microscopy, scanning electron microscopy and spectroscopic analysis." (4, p. 56)

Although Reference (4) indicated that technical reports were expected which would further describe experimental results, Reference (7) reported that documents which detailed the final mirror evaluation were not published. It was briefly reported (Reference (7)): "The experiment successfully produced mirrors. The researchers found no significant difference in the quality or properties of the 'space made' mirrors from the 'ground-made' samples. Due to research budget constraints, no final reports were issued by the research labs."

Key Words: Crystal Growth From Vapor, Vacuum Film Deposition, Vapor Deposition, Vaporization, Sublimation, Vapor/Solid Interface, Coated Surfaces, Films, Thin Films, Metals, Source Material, Substrates, Direct Exposure to Space Environment, Vacuum, Space Vacuum, Outgassing, Contamination Source, Oxidation, Mirror Production, Optics Applications, Optical Transmission, Sample Not Processed As Planned, Rocket Vibration, Payload Survivability, Deterioration of Samples After Zero-G Flight

Number of Samples: six
Sample Materials: Five of the tubes were configured with a tungsten filament coated with a metallic element. Of these five tubes, two employed aluminum as the element, two employed gold as the element, and one employed silver as the element. The sixth (hour-glass shape) tube was configured to process sodium.
(W*, Al*, Au*, Ag*, Na*)

Container Materials: Five tubes dedicated to metallic elements: steel; one tube dedicated to sodium: glass

Experiment/Material Applications:
"...the need for high-quality mirrors for such applications as lasers and telescopes which operate in the lower UV spectrum" (4, p. 49) were cited as motivation for exploring the mirror production.

"...in using a vapor deposition technique in the microgravity environment of space, a more uniform distribution of a reflective coating on a glass substrate could be achieved than a possible on Earth." (5, p. 32)

"The placement of the filaments and the weights of the elements on them were selected... to achieve the desired variety of film thicknesses coating the quartz lenses. To cover the lower UV spectrum, gold, silver and aluminum were selected as elements to put on the filaments in the steel tubes.... Sodium was selected as it was an interesting element to study." (4, p. 52)

References/Applicable Publications:
(1) Cargo Systems Manual: GAS Annex for STS 61-B, JSC-17645 61-B, p. 28-9, September 24, 1985. (short description; preflight)

(2) Mirror Experiment Wins Telesat Space Competition. Telesat Canada News Release, Personnel and Public Affairs Division, January 18, 1984. (preflight)

(3) Covault, C.: Astronauts Deploy Commercial Payloads, Ready Structures for Space Assembly. AW&ST, December 2, 1985. (post-flight)

(4) Hoffer, D.: Towards a Better Mirror. In Goddard Space Flight Center's 1986 Get Away Special Experimenter's Symposium, October 7-8, 1986, pp. 49-57, NASA CP-2438. (post-flight)

(5) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA GAS Team, 1989, p. 32. (post-flight; very brief description)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(7) Input received from Co-Investigator D. Hoffer, August 1993.

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CHAPTER 11

DIFFUSION

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Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt. Date: September 1973
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: Transparent, plastic, forcepts container filled 3/4 of the way with water
Builder of Processing Facility: Not applicable (the facility was merely a piece of onboard equipment).

Experiment:
Diffusion in Liquids (SD15, TV115)

On Earth, density differences within a liquid system often result in convective mixing of the fluid. This gravity-induced convection, coupled with gravity-independent diffusion, contributes to the overall mass transfer within the system. In space, the convective contribution is greatly reduced and a closer examination of the diffusion contribution can be observed.

This Skylab science demonstration was the first in a series of investigations designed by Facemire et al. to study low-gravity diffusive mass transfer. The specific objective of the demonstration was to photographically document the space diffusion of tea in water.

In preparation for the experiment, Skylab pilot Jack Lousma filled a 1/2 inch diameter, 6-inch-long transparent tube three-fourths of the way full with water. A highly concentrated tea solution was then delivered to the water surface (via a 5 cc syringe) through a synthetic fiber wad. The tube was then capped. (The fiber pad was employed to try to bring the tea and water in contact without entrapped air.)

Three different attempts to produce an even distribution of the tea through the wad were unsuccessful. During a fourth (wad) attempt, "a good bubble free interface" was realized. The next day, Lousma reported that no diffusion of the tea in the liquid had occurred. Thus, the experiment was initiated again.

During this new experimental run, the wad was removed and the tea was delivered directly on top of the water. After an air bubble between the tea and water was removed via the syringe, a "smooth, continuous interface" was achieved. The tea was allowed to diffuse during the next 3 days.

Post-flight, 16 mm photographs of the diffusion were analyzed. Reportedly, "Although the camera was slightly out of focus, it was evident on the film that diffusion occurred in the... system. In 51.5 hr., the visible diffusion front advanced 1.96 cm." (6, p. 2) It was noted that the diffusion front became increasingly parabolic during the demonstration. It was surmised that "...very little diffusion occurred near the... [container] wall. This means that some retarding force, not usually seen on earth, was present; this force... [was] hypothesized to be electrostatic repulsion." (6, p. 4)

<Note: While References (4), (6) and (9) indicate diffusion occurred during 51.5 hours, References (2) and (3) indicate diffusion occurred during 45 hours. The Principal Investigator reported that to the best of her knowledge, "...the 51.5 hr is correct for the μg diffusion and 45 hr was the ground test." (8)>

A similar, ground-based control experiment was performed for comparison to the space investigation. "After 45.5 hr[.], three distinct zones of tea were visible: (1) a dark area, (2) an area of medium darkness, and (3) a very light area. The very light area would probably not have been apparent on the out-of-focus film if it were present in the flight test. Therefore, the advance of the second area was used for comparison to the flight. This medium-colored area [1-g] had advanced 1.6 cm in 45.5 hr. The effects of convection on the ground test (possibly the very light area was caused by convective mixing) make absolute comparison of the zero-g case impossible." (6, p. 2)

A one-dimensional analysis of the predicted rate of advance of the diffusion front in the Skylab environment was made (see Reference (9)). "From refractive index measurements of various tea solutions, it was determined that C/C_o [concentration at front/original concentration (1.0)] in the range of 0.075 +/- 0.03 was likely to be visible on the film." (9, p. 5) Thus, "...by using the diffusion coefficient of sugar in water and by assuming that the determination of the visible concentration to be 0.075 +/- 0.03 is correct, then in 50 hours it would be expected that the tea should advance 1.77 +/- 0.4 cm. This value is very close to the actual value of 1.96 cm." (9, p. 9)

Key Words: Diffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Mass Transfer, Diffusion Coefficient, Diffusion Boundary, Aqueous Solutions, Buoyancy-Driven Convection, Solutal Gradients, Density Difference, Liquid/Liquid Interface, Interface Shapes, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Bubbles, Electrostatic Effects, Wall Effect, Photographic Difficulties

Number of Samples: One experimental setup was used four times during the mission.
Sample Materials: Tea (with sugar) at a concentration approximately seven times that of normal drinking tea; water
Container Materials: plastic

Experiment/Material Applications:
Diffusion in liquids was the first Skylab science demonstration and was devised in response to a request from the crew for meaningful hands-on research.

Diffusion is a fundamental mass transfer process of importance to many fluids and materials science disciplines including solidification processes.

References/Applicable Publications:
(1) "Diffusion in Liquids." In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-87. (post-flight results)

(2) Naumann, R. J. and Herring, H. W.: Diffusion in Liquids Demonstration. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, p. 86. (short summary)

(3) Bannister, T. C.: Science Demonstrations on Skylab in the Material Processing Area. In Proc. Third Space Processing Symposium, Skylab Results, Vol. 1, April 30-May 1, 1974, MSFC, p. 491.

(4) "TV115 - Diffusion in Liquids." In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-47 - 7-49. (post-flight)

(5) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

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(8) Input received from Principal Investigator B. Facemire, July 1993.

(9) MSFC Science Demonstrations Performed by Pilot J. Lousma on Skylab 3. Quick-Look Report, November 5, 1973, NASA Marshall Space Flight Center, Space Sciences Laboratory, 16 pp.

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Co-Investigator(s): Bannister, T. (2)
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Experiment Origin: USA
Mission: Skylab, SL-4, Third Skylab Manned Mission
Launch Date/Expt. Date: Although this mission took place during November 1973-February 1974, this experiment was not performed because of insufficient crew time.
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Science Demonstration, Skylab Manned Environment
Processing Facility: Unknown
Builder of Processing Facility: Not Applicable

Experiment:
Diffusion in Liquids (SD15, TV115)

This Skylab science demonstration was the second in a series of investigations designed by Facemire et al. to study low-gravity diffusive mass transfer (see Facemire, SL-3, "Diffusion of Liquid"). The specific objective of the experiment was similar to the earlier SL-3 diffusion demonstration: to photographically document the low-gravity diffusion of tea in water. The experiment goals were extended on SL-4 to include (1) obtaining focused photographs of the diffusion front and (2) testing "the hypothesis that electrostatic repulsion between tea particles and the plastic wall impeded diffusion along the wall." (1, p. 7-48)

Reportedly, the experiment was not performed because of a lack of available crew time.

No further information concerning this experiment could be located at this time.

Key Words: Diffusion, Liquid/Liquid Diffusion, Double Diffusion, Diffusive Mass Transfer, Mass Transfer, Diffusion Coefficient, Diffusion Boundary, Aqueous Solutions, Buoyancy-Driven Convection, Solutal Gradients, Density Difference, Liquid/Liquid Interface, Liquid Transfer, Bubbles, Electrostatic Effects, Wall Effect, Sample Not Processed As Planned

Number of Samples: not applicable
Sample Materials: not applicable
Container Materials: not applicable

Experiment/Material Applications:
See Facemire, SL-3, "Diffusion of Liquids"

References/Applicable Publications:
(1) "TV115/Diffusion in Liquids". In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA Technical Memorandum NASA TM X-64820, September 1974, pp. 7-47 - 7-49. (post-flight)

(2) Input Received from Principal Investigator B. Facemire, July 1993.

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Co-Investigator(s): Unknown
Affiliation(s): (1) During Skylab: Howard University, Washington, DC, Currently: Unknown

Experiment Origin: USA
Mission: Skylab, SL-3, Second Skylab Manned Mission
Launch Date/Expt Date: September 1973 (Month experiment was completed)
Launched From: NASA Kennedy Space Center, Florida
Payload Type: Materials Processing Facility (MPF) panels located forward from the Multiple Docking Apparatus (MDA) area, Skylab Manned Environment
Processing Facility: Multipurpose Electric Furnace System (MEFS)
Builder of Processing Facility: Westinghouse Astronuclear Laboratory, Large, Pennsylvania

Experiment:
Radioactive Tracer Diffusion (M558)

This Skylab SL-3 experiment was designed to investigate the low-gravity, self diffusion of radioactive liquid zinc-65 in non-radioactive liquid zinc. The specific objectives of the investigation were to (1) determine the experimental diffusion coefficients for liquid zinc (when the gravity-induced convective contributions are reduced) and (2) estimate the amount of reduction of gravity-induced convection in space.

Prior to the Skylab mission, three zinc/zinc-65 samples (designated as 'A-6', 'A-7', and 'B-5') were prepared. Samples A-6 and A-7 each consisted of a pure zinc bar (0.286 inch diameter, 1.592 inches long) which had been cold welded to a zinc-65 pellet (0.250 inch long). Sample B-5 consisted of a zinc-65 pellet (0.250 inch long) sandwiched (and cold welded) between two pure zinc bars (each 0.796 inch long). Each sample was inserted into its own carbon tube and sealed with carbon end caps. Each sample/carbon tube assembly was placed in a tantalum tube. Tantalum plugs were welded on each end under an argon atmosphere. Finally, these sealed, tantalum tubes/ampoules were leak tested by immersion in an ethylene glycol bath at 100 ^{o}C.

Prior to the initiation of the space experiment, each specimen ampoule was placed in the Skylab M518 Multipurpose Electric Furnace. Sample A-6 was configured in the tantalum tube such that its zinc-65 pellet was located in the cold end of the furnace. Sample A-7 was configured such that the zinc-65 pellet was located in the hot end of the furnace. Sample B-5 was configured such that the radioactive pellet was located between the hot and cold ends of the furnace.

During the Skylab SL-3 experiment, the zinc samples were subjected to a thermal soak of 775 ^{o}C for 1 hour (see Reference (1) for complete thermal history). Two sets of three samples each were processed on Earth for comparison: one set was soaked at 775 ^{o}C for 2 hours and the second set was soaked at 775 ^{o}C for 1 hour.

Post-flight examination of all the low-gravity processed samples indicated unique wrinkled surfaces in those sections located in the hot end of the furnace. This characteristic was attributed to (1) solidification shrinkage, and/or (2) a surface effect occurring during heating, and/or (3) higher heat removal at certain sites along the cylinder. Reportedly, "Ground based samples became shorter and thicker after melting because of the filling of space provided for expansion." (1, p. 437) However, none of the 1-g samples exhibited the surface wrinkles. It was reported that all 1-g and ground-based samples weighed less after processing (probably because of zinc evaporation).

A low-level gamma-ray spectrometer was used for determining gamma intensity from the zinc samples (see Reference (1) for procedure). All ground-based samples indicated a complete mixing of the zinc-65. Distribution predictions (using a pure diffusion mechanism) revealed a uniform distribution would be expected after (1) 73.5 hours for the zinc-65 pellet located on one end (A-type) and (2) 18.38 hours for the pellet located in the middle (B-type). However, a uniform distribution was achieved in all ground-based samples after a diffusion time of less than 3 hours.

The zinc-65 distribution in the Skylab samples was reported to be a "textbook" example of the solution of a one-dimensional, time-dependent diffusion equation (Fick's law). The radioactive zinc "...concentration gradient supports the observation that liquid metal diffusion was the main mechanism of transport. The plot of experimental data for each of the Skylab samples was matched to a remarkable degree with a plot from the solution of the pure diffusion equation under linear temperature conditions...." (1, p. 438) The average diffusion coefficient determined from the Skylab samples (at 550 ^{o}C) was 4.28×10^{-5} cm^{2}/sec. This value was 50 times less than the reported 1-g diffusion coefficient.

Considering the Skylab coefficient as convection free, the magnitude of the convective velocity from the 1-g samples was determined to be 4.16×10^{-4} cm/sec. Therefore, it was concluded that very small convective velocities can significantly affect the diffusion of impurities in liquid zinc. From a plot of the diffusion coefficient (ln D) versus 1/T, the equation of the self-diffusion coefficient for the Skylab samples was determined to be:

$D = 9.17 \times 10^{-4} e^{-5160/RT} cm^2/sec$
for 693 K (420 ^oC) < T < 973 K (700 ^oC)

where T is in degree Kelvin and R is 1.987 cal/(mol-K).

Key Words: Diffusion, Radioactive Tracer Diffusion, Self-Diffusion, Interdiffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Diffusion Coefficient, Fick's Law, Tracer Particles, Melt and Solidification, Thermal Soak, Heat and Mass Transfer, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Concentration Distribution, Surface Morphology, Sample Shrinkage, Sample Detachment From Crucible, Sample Evaporation, Impurities

Number of Samples: three
Sample Materials: Pure zinc with pellets of radioactive zinc-65 tracer
(Zn*)
Container Materials: Graphite tubes contained in tantulum ampoules
(C*, Ta*)

Experiment/Material Applications:
Many low-gravity fluids and materials processing experiments hope to attain purely diffusive mass transport (rather than convective and diffusive mass transport). This experiment illustrated that convection is greatly reduced in the low-gravity environment.

References/Applicable Publications:
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(3) Larson, D. J., Jr.: Metallurgical Analysis of Skylab M 552 and M 558 Samples, Final Report, 1978, Grumman Research Dept., Report RE-565. (post-flight)

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(7) Experiment M558-Radioactive Tracer Diffusion. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-62 - 5-65. (post-flight)

(8) Braski, D. N., O'Donnell, F. R., and Kobisk, E. H.: Progress Report on Specimen Fabrication and Ground Tests for NASA Skylab Experiment M-558. Oak Ridge National Laboratory, ORNL-TM-4152, April 1973, 40 pp. (preflight)

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(10) Multipurpose Electric Furnace (M518). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-46 - 12-49. (processing facility)

(11) Radioactive Tracer Diffusion (M558). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-50. (post-flight; very short summary)

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Co-Investigator(s): None
Affiliation(s): (1,2,3) Technische Universität Clausthal, Germany

Experiment Origin: Federal Republic of Germany
Mission: TEXUS 1
Launch Date/Expt. Date: December 1977
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal four-chamber furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Reaction Kinetics in Glass Melts

The production of glasses on Earth involves many different reaction mechanisms: (1) the dissolution of quartz grains in glass melts, (2) the refining of the glass melt (fining procedures), and (3) the corrosion of refractory material by the melt. These mechanisms are controlled by buoyancy-driven convection and mass diffusion within the melt.

Terrestrial diffusion experiments of glass melts with low viscosities illustrate that convective disturbances are always present, even if the experiments are performed in capillary systems. "In particular buoyancy convection superimposes diffusive mass transport; therefore only apparent diffusion coefficients can be obtained." (10, p. 2122) In a reduced gravity environment, buoyancy-driven convection in such melts will be reduced, and larger scale experimental systems can be employed.

This TEXUS 1 experiment was the first in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses. (The series of experiments as a whole included studies in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objective of this experiment was to investigate diffusion phenomena within a glass melt under low-gravity conditions.

Prior to the mission, one sample, comprised of two cylinders (each 12 mm diameter, 10 mm long) was prepared. The first cylinder was comprised of Na_2O . 3 SiO_2 and the second of Rb_2O . 3 SiO_2. One end of each cylinder was polished, and then the cylinders were joined together at the polished surfaces. The joined material was enveloped in platinum and placed in a Mo

crucible. (See Reference (5) for complete, preflight sample preparation procedures.)

Prior to launch, the sample was heated in the TEXUS Experiment Module TEM 01-1 to a temperature above its glass transition point (550 ^{o}C). Once low-gravity conditions had been achieved, the sample was heated to 1160 ^{o}C (as registered at the middle of the sample) resulting in an effective reaction time of about 135 seconds. Reportedly, a "...step at the edge between both glass cylinders has the function to maintain an even interface..." at gravity levels approaching zero. (1, p. 264) Prior to the end of the low-gravity period, the sample was rapidly cooled.

A reference sample was processed on Earth using the same thermal profile. Post-flight comparison of the 1-g and low-gravity processed sample indicated that the low-gravity material had a more even interface. A slight radius of curvature at the interface of the low-gravity sample was observed and attributed to gas bubbles which formed during cooling.

Microprobe analysis was used to determine Na and Rb concentration profiles in the 1-g and low-gravity samples. While the 1-g sample profiles appeared to be strongly influenced by convection, the low-gravity sample profiles exhibited "...an undisturbed ion exchange process of Na^{+} and Rb^{+} ions through the interface between both glass melts." (1, p. 265)

The Na and Rb concentration profiles from the low-gravity sample were also used to obtain the binary chemical interdiffusion coefficient (see Reference (1) for details). A plot of this term versus Rb_2O concentration indicated (1) a concentration dependence and (2) a strong mixed-alkali effect. When the log of the interdiffusion coefficient was plotted versus 1/T(temperature) in an Arrhenius type graph, it was determined that (for a temperature range of 700 to 1160 ^{o}C) (1) $D_o = 5 \times 10^{-3}$ cm^2/s and (2) the activation energy was 75 kJ/mole.

For the glass of composition 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2 the following interdiffusion coefficient was found: $D_{Na,Rb} = 9.1 \times 10^{-6}$ cm^2/s.

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Melt and Solidification, Diffusive Mass Transfer, Diffusion Coefficient, Interdiffusion, Liquid/Liquid Diffusion, Double Diffusion, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Ion Exchange, Concentration Distribution, Reaction Kinetics, Bubbles, Bubble Formation, Interface Shapes, Interfacial Curvature, Solid/Liquid Interface, Liquid/Liquid Interface, Quench Process

Number of Samples: one
Sample Materials: Quasi-binary Na_2O . 3 SiO_2 - Rb_2O . 3 SiO_2 (Na*O*Si*O*, Rb*O*Si*O*, Na*O*Si*O*Rb*O*Si*O*)
Container Materials: Platinum surrounded by molybdenum (Pt*, Mo*)

Experiment/Material Applications:
See **Experiment** section (above) and Frischat, TEXUS 13 (this chapter).

References/Applicable Publications:
(1) Herr, K., Barklage-Hilgefort, H. J., and Frischat, G. H.: Reactions Between Glass Melts. In Proc. of the 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142 (June 1979), pp. 263-266. (post-flight)

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(4) Herr, K. and Frischat, G. H.: Ion Exchange Between Glass Melts of the system Na2O-Rb2O-SiO2. J. Non-Cryst. Solids, Vol. 41, (1980), pp. 117-126.

(5) Frischat, G. H., Braedt, M., and Beier, W.: Reactions in Glass Melts. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 161-165. (post-flight TEXUS 1,4,6)

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(8) Input received from Experiment Investigator, August 1988 and July 1993.

(9) Reaction Kinetics in Glasses. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 120-121. (post-flight)

(10) Meier, M., Braetsch, V., and Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. J. Am. Ceram. Soc., Vol. 73(7), 1990, pp. 2122-2123. (concentrates on results from later flights)

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 4
Launch Date/Expt. Date: May 1981
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal four-chamber furnace (same furnace used during TEXUS 1)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Reaction Kinetics in Glass Melts: Corrosion of Silica

This TEXUS 4 experiment was the second in a series of investigations designed by Frishat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1). (The series of experiments as a whole included studies in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objective of this experiment was to investigate the corrosion of vitreous silica by two sodium silicate glass melts under low-gravity conditions.

Prior to the flight, two experimental samples were prepared. In the first sample, a Type III vitreous silica glass cylinder was sandwiched between two cylinders of Na_2O . 3 SiO_2 (25 mol% Na_2O). In the second sample, a Type III vitreous silica glass cylinder was sandwiched between two cylinders of Na_2O . 2 SiO_2 (33 mol% Na_2O). As part of the preparation procedure, the sandwiched samples were place in cylindrical, Pt jackets and sintered at 550 ^oC in a vacuum furnace. The cylinders were then closed by Pt caps and the entire configuration sealed under vacuum in a Ta-Zr-Mo cartridge. (See Reference (4) for additional sample preparation details.)

Just prior to liftoff, the samples were preheated to 700 ^oC in the TEXUS Experiment Module TEM 01 Furnace (maximum heating rate of 0.5 K/s). After launch, the material was heated at a maximum heating rate of 3.5 K/s until the effective reaction temperature of 1420 ^oC was achieved. "...the sample was held at that temperature for about 3 min and was then cooled at a rate of 20 K/s by flushing with He. Cooling near the transformation temperatures of the sodium silicate glasses took place more slowly...." (4, p. 2) An effective reaction time of 182 seconds was es-

timated. (The hold time and cooling were achieved during the low-gravity phases of the rocket flight.) Reference samples were prepared in the same manner on Earth using an identical time-temperature profile.

After sample recovery and prior to analysis, the specimens were heated to 550 ^{o}C (above the glass transition temperature, T_g) and slowly cooled to room temperature. This procedure was performed to minimize mechanical stress in the samples.

Post-flight analysis of the specimens included (1) electron microprobe studies (for determination of the corrosion profiles) and (2) photography (after axial slicing, grinding, and polishing). Reportedly, the Earth-processed samples contained clearly visible convective vortices which were absent in the low-gravity processed material. The 1-g processed samples (because of the presence of convection) also exhibited an apparent inter-diffusion coefficient which was smaller than that seen for the low-gravity samples. (Reference (1) reported that this result was also observed in the TEXUS 1 low-gravity sample and the corresponding Earth-processed material (see Frischat, TEXUS 1 (this chapter)).) The TEXUS 4 low-gravity processed samples demonstrated the expected undisturbed diffusion profiles. The interdiffusion coefficients for the 1-g and low-gravity processed samples were reported as:

For 25 mol% Na_2O material:
low-gravity sample: (2.8 +/- 0.5) x 10^{-7} cm^2/s
1-g sample: (1.8 +/- 0.5) x 10^{-7} cm^2/s
1-g, capillary experiment sample: 2.2 x 10^{-7} cm^2/s

For 33 mol% Na_2O material:
low-gravity sample: (6 +/- 1) x 10^{-7} cm^2/s
1-g sample: (3 +/- 1) x 10^{-7} cm^2/s

All diffusion coefficient values are valid for T = 1420 ^{o}C.

Reportedly, "...the values obtained under microgravity can be regarded as intrinsic data, while the coefficients obtained under normal gravity are apparent quantities only." (4, p. 4) (See Reference (4) for details.)

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Model Materials, Melt and Solidification, Corrosion, Diffusive Mass Transfer, Diffusion Coefficient, Interdiffusion, Liquid/Liquid Diffusion, Double Diffusion, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Reaction Kinetics, Solid/Liquid Interface, Liquid/Liquid Interface, Quench Process, Vacuum

Number of Samples: two
Sample Materials: (1) Type III vitreous silica sandwiched between Na_2O . 2 SiO_2 (sodium disilicate) and (2) Type III vitreous silica sandwiched between Na_2O . 3 SiO_2 (sodium trisilicate)
(Na*O*Si*O*)
Container Materials: Cylindrical platinum jacket sealed in a tantalum-zirconium-molybdenum cartridge
(Pt*, Ta*Zr*Mo*)

Experiment/Material Applications:
"Na_2 . 2 SiO_2 and Na_2O . 3 SiO_2 are model glasses for the alkaline silicate glass system. There was no difference between quartz crystal and vitreous silica during earlier experiments, thus vitreous silica is a model material for fused silica refractoring." (Reference (5))

See also Frischat, TEXUS 1, **Experiment** section (this chapter).

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 6
Launch Date/Expt. Date: May 1982
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: MBB/ERNO, Bremen, Germany

Experiment:
Reaction Kinetics in Glass Melts

This TEXUS 6 experiment was the third in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4). (The series of experiments, as a whole, included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objective of this experiment was to investigate the self diffusion of sodium in two different glass melts.

Prior to flight, two samples were prepared: (1) Na_2O . 3 SiO_2 (designated glass 1) and (2) 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2 (designated glass 3). Radioactive Na^{22} was included in each sample as a tracer for determining the self diffusion coefficients. (See Reference (1) for sample preparation details.)

During the mission, the TEXUS Experiment Module TEM 01-2 was used to process the materials. Both samples were processed at a self diffusion temperature of 1200 ^{o}C. Corresponding samples were processed on Earth for comparison.

Post-flight examination of the specimens revealed that the interface of the low-gravity samples remained planar while the interface of the 1-g samples was distorted by convective flows. The following values for the self-diffusion coefficient, D^*_{Na}, were obtained using a modified residual activity method:

For glass 1:
$(2.75 \pm 0.4) \times 10^{-5}$ cm^2/s low-gravity
$(5.35 \pm 0.5) \times 10^{-5}$ cm^2/s 1-g

For glass 3:
$(2.5 \pm 0.6) \times 10^{-5}$ cm^2/s low-gravity
$(3.4 \pm 0.4) \times 10^{-5}$ cm^2/s 1-g

Reportedly, (1) under normal gravity conditions, density-induced convection influenced the diffusion process and lead to an unrealistic D* value, and (2) for the chosen temperature interval, the mixed alkali effect was minimized so that the diffusion coefficients in Melt 1 and Melt 3 were nearly the same.

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Melt and Solidification, Diffusive Mass Transfer, Radioactive Tracer Diffusion, Self-Diffusion, Diffusion Coefficient, Tracer Particles, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Concentration Distribution, Reaction Kinetics, Interface Shapes, Planar Solidification Interface,Interfacial Curvature, Solid/Liquid Interface

Number of Samples: two
Sample Materials: (1) Sodium Trisilicate, Na_2O . 3 SiO_2 with radioactive ^{22}Na tracer and (2) 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2 with radioactive ^{22}Na tracer
(Na*Si*O*, Na*O*Rb*Si*O*)
Container Materials: platinum
(Pt*)

Experiment/Material Applications:
See Frischat, TEXUS 1, **Experiment** section and Frischat, TEXUS 13 (both in this chapter).

References/Applicable Publications:
(1) Frischat, G. H., Braedt, M., and Beier, W.: Reactions in Glass Melt Systems. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 161-165.

(2) Input received from Experiment Investigator, August 1988 and August 1993.

(3) Frischat, G. H.: Microgravity Research in the Field of Glasses and Ceramics. In Symposium on Industrial Activity in Space, Stresa, Italy, May 2-4, 1984, Proceedings, Paris, Eurospace, 1984, pp. 128-136.

(4) Frischat, G. H. and Braedt, M.: Reactionskinetik in Glasschmelzen, Natrium-Selbstdiffusion in Alkalisilicatschmelzen -Projekt TEXUS VI. BMFT-Forschungsbericht 01 QV 467-ZA/ANA/ALN/7773-1.9, February 1983. (in German)

(5) Braedt, M. and Frischat, G. H.: Sodium Self Diffusion in Alkali Silicate Glass Melts as Obtained by a μg Experiment. J. Amer. Ceram. Soc., 67 (1984), C54-C56.

(6) Reaction Kinetics in Glass Melts: Sodium Self-Diffusion in Alkali Silicate Melts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 124-125. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Reaction Kinetics in Glass Melts (ES307A and ES307B)

This Spacelab 1 experiment was the fourth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6). (The series of experiments, as a whole, included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objectives of this experiment were to (1) investigate the interdiffusion between Na_2O . 3 SiO_2 and Rb_2O . 3 SiO_2 glass melts and (2) determine the corrosion of SiO_2 glass by different alkali-silicate melts.

During the Spacelab 1 mission, the Isothermal Heating Facility (IHF) was used to process three samples each comprised of either two or three cylinders of different materials stacked one on top of another. The cylindrical materials (80 mm total height) were contained in a single cartridge. The cylinders, as stacked from top to bottom, were (1) Na-silicate glass (8 mm), (2) SiO_2 (4 mm), (3) Na-silicate glass (8 mm), (4) Na_2O . 3 SiO_2 (20 mm), (5) Rb_2O . 3 SiO_2 (20 mm), (6) Rb-Silicate glass (8 mm), (7) SiO_2 (4 mm), and (8) Rb-Silicate glass (8 mm). Cylinders (1), (2), (3) (comprising one sample) and cylinders (6), (7), and (8) (comprising a second sample) were used for the corrosion portion of the experiment (designated as ES 307B). Cylinders (4) and (5) (comprising the third sample) were used for the interdiffusion experiment (designated as ES 307A).

Reportedly, the IHF malfunctioned during the experiment because of "...the misworking vacuum gas system of the MSDR [Material Science Double Rack]. An undefined gas entry outside the IHF caused the closing of an IHF vent. Heating continued and a

critical pressure in the IHF was reached after a few minutes, forcing the IHF to... [fail]. This was verified during the 1g reference test." (1, p. 110)

Because of a failure of the IHF heating system, "...only ES 307A ran in part, whereas ES 307B was not processed at all. Moreover, the temperature-time program, which was planned to maintain a 30 min period of constant temperature of 1200 ^{o}C was quite different." (1, p. 109) As a consequence, only the interdiffusion experiment could be evaluated.

After the Spacelab 1 mission, the interdiffusion experiment sample was heat treated to minimize possible thermal stress. The material was then removed from the cartridge and placed in a water-free methanol bath to prevent corrosion by air. Prior to examination, the sample was cut axially and prepared for photographic and electron-microprobe analyses. Calculation of the effective diffusion time was performed using the formula of Makin et al. (see Reference (5)). The effective diffusion time, t_{eff}, was reported to be 1730 seconds with an effective temperature, T_{eff}, of 1180 ^{o}C. (A t_{eff} of 2170 s and a T_{eff} of 1200 ^{o}C had originally been planned.)

Measurements of the low-gravity concentration profiles (parallel and normal to the original phase boundary of the two glass cylinders) indicated an undisturbed interdiffusion process. In contrast, the 1-g processed sample profiles indicated a wavy form. Such profiles are typical of systems with diffusion and convection influences.

The low-gravity, interdiffusion coefficient for an Rb mole fraction of 0.5 was reported (Reference (4)) as (1.05 +/- 0.2) x 10^{-5} cm^2/s (T = 1180 ^{o}C). (See Reference (1) for details of interdiffusion coefficient analysis.) This value fits the order of data obtained during earlier low-gravity experiments (see Frischat, TEXUS 1). The value of the interdiffusion coefficient obtained from the 1-g reference experiment was greatly effected by convection effects.

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Model Materials, Melt and Solidification, Corrosion, Diffusive Mass Transfer, Diffusion Coefficient, Interdiffusion, Liquid/Liquid Diffusion, Double Diffusion, Buoyancy-Driven Convection, Concentration Distribution, Reaction Kinetics, Solid/Liquid Interface, Liquid/Liquid Interface, Thermal Environment More Extreme Than Predicted, Sample Not Processed As Planned, Hardware Malfunction, Furnace Malfunction

Number of Samples: Three samples comprised of several cylinders
Sample Materials: (1) For ES 307A, sodium trisilicate, $Na_2 . 3 SiO_2$ and $Rb_2 . 3 SiO_2$; (2) for corrosion experiments: $Na_2O . 2 SiO_2$, $Na_2O . 3 SiO_2$, $Rb_2O . 3 SiO_2$, and SiO_2 glass
(Na*Si*O*, Rb*Si*O*, Na*O*Si*O*, Rb*O*Si*O*, Si*O*)
Container Materials: Platinum
(Pt*)

Experiment/Material Applications:
See Frischat, TEXUS 1, **Experiment** section and Frischat, TEXUS 4 and TEXUS 13 (all in this chapter).

"The largest group of technologically applied glasses belongs to the alkaline silicate glasses and $Na_2O . 2 SiO_2$, $Na_2O . 3SiO_2$ and $Rb_2O . 3SiO_2$ are model materials for this glass melt.... Silica glass used for the corrosion experiments is a model for most of the silica containing solids." (4)

References/Applicable Publications:
(1) Braedt, M., Braetsch, V., and Frischat, G. H.: Interdiffusion in the Glass Melt System $(Na_2O + Rb_2O).3 SiO_2$. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA Publication ESA SP-222, pp. 109-112. (post-flight results)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurements and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab 1)

(3) Wittmann, K.: The Isothermal Heating Facility. In 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 49-54. (IHF facility)

(4) Input received from Experiment Investigator, August 1988 and August 1993.

(5) Makin, S. M., Rowe, A. H., and LeClair, A. D.: Self Diffusion in Gold. In Proc. Phys. Soc. London B, Vol. 10, 1957, pp. 545-552. (reference used in sample analysis)

(6) Frischat, G. H., Braedt, M., and Beier, W.: Reactions in Glass Melt Systems. In Proceedings of the 4th European Symposium on Materials Sciences under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 161-165. (pre-flight)

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #10, STS-011 (STS 41-B, Challenger)
Launch Date/Expt. Date: February 1984
Launched From: NASA Kennedy Space Center, Florida
Payload Type: West German Get Away Special (GAS) MAUS Canister DG-318
Volume of Canister: 5.0 cubic feet
Location of Canister: The West German Shuttle Pallet Satellite (SPAS-01A) (SPAS was a small experiment carrier removed from the STS payload bay by the Canadian Remote Manipulator Arm, but remained attached to the arm throughout the mission at a position overhead about 5 to 10 feet from the forward payload bay bulkhead. The satellite was returned to the cargo bay before the shuttle's return.)
Primary Developer/Sponsor of DG-318: The German Ministry of Research and Technology (Bundesministerium für Forschung und Technologie (BMFT))/Messerschmitt-Boelkow-Blohm (MBB-ERNO)
Processing Facility: Isothermal Furnace
Builder of Processing Facility: The experiment configuration was developed by the Principal Investigator.

Experiment:
Reaction Kinetics in Glass Melts (DG-318)

The removal of gas bubbles from the melt (fining) during a glass refining process is a problem which has been studied extensively in the past (see Reference (1) for details). Earlier work emphasized the importance of gas diffusion and gas solution during the fining process.

Bubble-radius versus time data has been used to determine diffusion coefficients during ground-based experiments. However, unwanted gravity-dependent convective effects disturb the concentration fields around the bubble and make the calculation of the diffusion coefficients inaccurate.

This STS-011 experiment was the fifth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1). (The series of experiments, as a whole, included examination in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objective of this experiment was to (1) investigate the shrinking of a helium gas

bubble in a glass melt and thus (2) determine the diffusion coefficient of helium in a glass melt.

The experiment was performed in an isothermal furnace configured within a MAUS container. (The container, which is similar to a Get Away Special (GAS) Canister, was part of the STS-011 Shuttle Pallet Satellite (SPAS 01A) system.) The furnace was equipped with sapphire windows which permitted photography of the shrinking bubble at 1 min intervals. Illumination for the photography was provided by an electronic flash.

During the investigation, a sodalime silica glass sample (30 mm in length, 16 mm in diameter) containing a He bubble was melted at 1100 ^{o}C. The total observation time was 93 min.

Information on the bubble radius versus time was attained from post-flight examination of the photographs. It was reported that the bubble shrank almost linearly with time to a residual volume of 10%. It was also reported that the bubble center remained at a fixed location during the experiment. From the radius versus time data, the intrinsic diffusion coefficient of He in the glass melt was determined (see Reference (1) for analysis details).

The low-gravity, time-temperature data indicated that the sample temperature was not constant through any portion of the experiment. "This was due to the restricted electrical power resources and probably an increase of the emissivity of the stainless... [steel heat] shields during several thermal tests. The initial objective to evaluate the the diffusion coefficient for a constant temperature had to be replaced by the... [calculation] of a temperature dependent one. This led to the values for the diffusion coefficient for two temperatures." (7)

The Principal Investigator's response (Reference (7)) reported that the calculated diffusion coefficients from the low gravity experiments were:

$D = 3.2 \times 10^{-5}$ cm^2/s (T = 936 ^{o}C)
$D = 5.0 \times 10^{-5}$ cm^2/s (T = 1036 ^{o}C)

<Note: The Experiment Investigator reported that the values of the diffusion coefficients as reported in Reference (1) were preliminary results. "A later detailed analysis showed, that it was only possible to evaluate about 30 minutes of the experiment. This was due to the applicated model, which had to take into account the variable temperature. This means a temperature range from 936^{o}C to 1036 ^{o}C." (7) The meaning of "applicated" in the above quote was unclear to the editors.>

The low-gravity results were compared to two earlier experiments performed on Earth (see Reference (6)). The diffusion coefficients for these ground-based studies were reported to be:

$D = 4.24 \times 10^{-5}$ cm^2/s ($T = 1086\ ^oC$)
$D = 8.08 \times 10^{-5}$ cm^2/s ($T = 1170\ ^oC$)

The time-temperature data from two ground-based experiments indicated that the temperature in each remained constant over a long period of time. This was because "...no batteries were used for the earthbound experiments. So there wasn't any problem with restricted electrical power." (7)

The differences in diffusion coefficients between the ground-based and low-gravity experiments were attributed to convective effects. "...it seems plausible that convection effects accelerate bubble shrinking so that a higher diffusion coefficient is simulated in the lg experiments, whereas a lower value is rather to be expected because of the lower helium solubility." (1, p. 357)

Reportedly, fitting the data from the Principal Investigator's response in an Arrhenius relation yielded:

Activation Energy $Q_2 = 56.6$ kJ/mol
$D_o = 8.95 \times 10^{-3}$ cm^2/s

Key Words: Diffusion, Glasses, Glass Melts, Binary Systems, Melt and Solidification, Diffusion Coefficient, Bubbles, Bubble Shrinkage, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Concentration Distribution, Reaction Kinetics, Liquid/Gas Interface, Thermal Environment More Extreme Than Predicted

Number of Samples: one
Sample Materials: sodalime silica glass with helium bubble (Si*O*, He*)
Container Materials: platinum (Pt*)

Experiment/Material Applications:
See Frischat, TEXUS 1, **Experiment** section (this chapter).

"The glass composition was chosen as a model glass to investigate gas diffusion in glass melts." (7)

References/Applicable Publications:
(1) Rosenkranz, V. and Frischat, G. H.: Shrinking of a Glass Melt Under Microgravity Conditions. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 353-357. (post-flight results)

(2) Frischat, G. H.: Microgravity Research in the Field of Glasses and Ceramics. In Symposium on Industrial Activity in Space, Stress, Italy, May 2-4, 1984, Paris, Eurospace, 1984, pp. 128-136. (very short summary of DG-318 and other microgravity efforts)

(3) Rosenkranz, B., Braetsch, B., and Frischat, G. H.: Gas Bubbles in Glass Melts Under Microgravity; I. Apparatus for Photographic Observation. Physics Chem. Glasses, 26 (1985), pp. 123-125.

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(6) Frischat, G. H. and Oel, H. J.: Bestimmung des Diffusionskoeffizienten von Helium in Glassschmelzen aus der Abnahme einer Blase, Glastechn. Ber., 38, pp. 156-166. (in German; results from earlier TEXUS experiments)

(7) Input received from Experiment Investigator, August 1988 and August 1993.

(8) Otto, G. H.: Experimental Results from Automated MAUS Payloads. In 39th Congress of the International Astronautical Federation, Bangalore, India, October 8-15, 1988, Paper Number IAF-88-351. (post-flight)

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics, Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (post-flight, GAS mission history)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 11
Launch Date/Expt. Date: April 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal four-chamber furnace (module used since TEXUS 1 but now with modifications in furnace design)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Reaction Kinetics in Glass Melts

This TEXUS 11 experiment was the sixth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011). (The series of experiments, as a whole included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objectives of the TEXUS 11 experiment were to (1) characterize the self- and interdiffusion in alkali silicate melts and (2) obtain results on diffusion behavior which would compliment earlier results obtained during the TEXUS 1, TEXUS 4, TEXUS 6 and Spacelab 1 experiments.

During the TEXUS 11 mission, two chambers of the isothermal four-chamber furnace were used (chamber TEM 01B and chamber TEM 01C). Each of these chambers held two samples: (1) a sample dedicated to studying the interdiffusion between glass melts Na_2O . 3 SiO_2 and Rb_2O . 3 SiO_2, and (2) a sample dedicated to studying self-diffusion in the glass melt 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2. During the low-gravity phase of the experiment, the samples in TEM 01B were held at a temperature of 1660 K for 110 seconds and the samples in TEM 01C were held at a temperature of 1545 K for 144 seconds. <Note: The samples and experimental procedures detailed above were reported by the Principal Investigator in 1993 and differed slightly from similar information reported in other applicable references.>

After the mission, the inter-diffusion samples were analyzed according to the Boltzman-Matano principle and the self-diffusion samples were analyzed using the residual activity technique. The

following results were reported (Reference (2)):

For 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2, the self-diffusion coefficient, D^*_{Na} = (2.6 +/- 1.3) x 10^{-5} cm^2/s at 1545 K and

= (4.7 +/- 0.4) X 10^{-5} cm^2/s at 1660 K.

The interdiffusion coefficient, $D^{\sim}_{Na,Rb}$ = (3.1 +/- 0.9) x 10^{-6} cm^2/s at 1545 K and

= 5.0 +/-0.5).5 X 10^{-6} cm^2/s at 1660 K.

<Note: The values above were reported by the Principal Investigator in 1993 and differed slightly from values reported in other references.>

Reportedly, the self diffusion data fit well into the "functionality" exhibited by earlier TEXUS (low-gravity) and capillary (ground based) experiments. "In contrast to the tracer diffusion coefficients, the inter-diffusion coefficients did not follow the Arrhenius law established on the basis of ground experiments using capillaries. Measurements of the specific electrical conductivity, for which the transfer model is analogous, showed also a clear deviation from the Arrhenius law, which was valid only at low temperature. Owing to the few experimental data available and to the inherent lack of accuracy of the results, it was not possible to give a satisfying explanation of this deviation." (6, p. 126)

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Melt and Solidification, Diffusive Mass Transfer, Diffusion Coefficient, Interdiffusion, Liquid/Liquid Diffusion, Double Diffusion, Self-Diffusion, Buoyancy-Driven Convection, Concentration Distribution, Density Difference, Reaction Kinetics, Solid/Liquid Interface, Liquid/Liquid Interface, Capillaries, Bioceramics, Optics Applications

Number of Samples: four
Sample Materials: (1) Na_2O . 3 SiO_2, Rb_2O . 3 SiO_2 and (2) 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2
(Na*O*Si*O*, Rb*O*Si*, Na*O*Rb*O*Si*O*)

Container Material: platinum surrounded by nickel (Pt*Ni*)

Experiment/Material Applications:
"An understanding of self- and inter-diffusion in alkali silicate melts is the basis for a theoretical description of transport processes in glass melts and is of fundamental technical and scientific relevance. Examples of such transport processes are the resolution of the batch in glass melts, corrosion of refectory material, homogenization and refinement processes like chemical solidification of glass or the production of optic light filaments and bioceramics.

"Since the transport phenomena are composed essentially of a diffusion [portion] and various convection portions which overlap, characteristic data is... [indispensable] to attribute them to individual technological processes. Density differences have the greatest influence on the convection term; they are caused by the earth's gravity. Under low-gravity conditions, this interfering factor can be kept negligible." (1, (translation))

See also Frischat, TEXUS 1, **Experiment** section and Frischat, TEXUS 13 (this chapter).

References/Applicable Publications:
(1) Frischat, G. H., Braetsch, V., and Meier, M.: Reaction Kinetics in Glass Melts. In TEXUS 11/12 Abschlussbericht, German Publication, H. Ahlborn (ed.), 1985. (in German)

(2) Input received from Experiment Investigator, August 1988 and 1993.

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(5) Meier, M., Braetsch, V., and Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. In Journal of the American Ceramic Society, Vol. 73, Number 7, 1990, pp. 2122-2123. (post-flight)

(6) Self- and Interdiffusion in Alkali-Silicate Melts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 126-129. (post-flight)

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 12
Launch Date/Expt. Date: May 1985
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2: isothermal four-chamber furnace (module used since TEXUS 1 but now with modifications in furnace design)
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Reaction Kinetics in Glasses

This TEXUS 12 experiment was the seventh in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011, TEXUS 11). (The series of experiments, as a whole, included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objectives of the TEXUS 12 experiment were to (1) characterize the self- and interdiffusion in alkali silicate melts and (2) obtain results on diffusion behavior which would compliment earlier results obtained during the TEXUS 1, TEXUS 4, TEXUS 6, TEXUS 11, and Spacelab 1 experiments.

One chamber of the isothermal four-chamber furnace was used for this experiment. The chamber contained (1) a sample for the interdiffusion experiment (Na_2O . 3 SiO_2. 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2) and (2) a sample for the self-diffusion experiment (0.75 Na_2O . 0.25 Rb_2O . 3 SiO_2. Reportedly (Reference (2)), "One nickel cartridge enclosed two platinum cartridges. The one platinum cartridge consisted of two platinum crucibles, the first with sodium silicate glass, the second was filled with the mixed alkaline glass; the leveled tops of the different glasses were pressed together. The other part of the cartridge is composed of two platinum crucibles, each of those contained one sample of glass. The tops of these glasses were coated with a thin layer of Na^{22} tracer. Afterwards the two samples were combined in a sandwich like construction." The samples were heated to 1610 K under low-gravity conditions.

Reportedly (Reference (2)), the following results were obtained:

For 0.75 Na_2O . 0.25 Rb_2O . 3 SiO_2 and T = 1610 K:
D^*_{Na} (self-diffusion coefficient) = (2.7 +/- 0.7) x 10^{-5} cm^2/s

Reportedly, the self-diffusion data fit well into the "functionality" resulting from earlier TEXUS (low-gravity) and capillary (ground based) experiments.

The obtained value for D~ (interdiffusion coefficient) from the diffusion couple Na_2O . 3 SiO_2 - 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2 was inconsistent to other D~-data.

No other information concerning this experiment could be located at this time.

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Model Materials, Melt and Solidification, Diffusive Mass Transfer, Diffusion Coefficient, Interdiffusion, Liquid/Liquid Diffusion, Double Diffusion, Self-Diffusion, Radioactive Tracer Diffusion, Tracer Particles, Heat and Mass Transfer, Buoyancy-Driven Convection, Concentration Distribution, Density Distribution, Reaction Kinetics, Solid/Liquid Interface, Liquid/Liquid Interface, Coated Surfaces, Capillaries, Optics Applications

Number of Samples: two
Sample Materials: Na_2O . 3 SiO_2 - 0.5 Na_2O . 0.5 Rb_2O . 3 SiO_2 and 0.75 Na_2O . 0.25 Rb_2O . 3 SiO_2. <Note: Although it was not specifically stated, it appears the Na tracer employed was radioactive.>
(Na*O*SiO*, Na*O*Rb*O*Si*O*)
Container Materials: platinum surrounded by nickel
(Pt*Ni*)

Experiment/Material Applications:
See Frischat, TEXUS 1, **Experiment** section and Frischat, TEXUS 13 (this chapter).

References/Applicable Publications:

(1) Frischat, G. H., Braetsch, V., and Meier, M.: Reaction Kinetics in Glass Melts. In TEXUS 11/12 Abschlussbericht, H. Ahlborn (ed.), 1985. (in German)

(2) Input received from Experiment Investigator, August 1988 and August 1993.

(3) Frischat, G. H., Braetsch, V., and Meier, M.: Reaction Kinetics in Glass Melts-- Self-and Inter-Diffusion in Alkali Silicate Melts. TEXUS 11/12 Rocket Preprogram, BMFT Research Report 01-QV-334 A5, October 1985.

(4) Zarzycki, J., Frischat, G. H., and Herlach, H. M.: Glasses. In Fluid Science and Materials Science in Space, Edited by H. U. Walter, Berlin, Heidelberg, New York: Springer, 1987, pp. 599-636.

(5) Meier, M., Braetsch, V., and Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. Journal of the American Ceramic Society, Vol. 73, Number 7, 1990, pp. 2122-2123. (post-flight)

(6) Self- and Interdiffusion in Alkali-Silicate Melts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 126-129. (post-flight)

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Material Science Double Rack (MSDR)
Processing Facility: Isothermal Heating Facility (IHF) furnace
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Homogeneity of Glasses - Measuring the Differences in Glass Crystallization Between Micro-g and 1-g (WL-IHF 05)

During the solidification of a glass, the melt freezes without significant crystallization. However, in a glass-ceramic material, partial or total crystallization occurs. At times, nucleating agents may be added to control the crystallization process. If only homogeneous nucleation occurs in the melt, then the prospect for glass formation is good. However, heterogeneous nuclei are easily formed at the container wall. Gravity-induced convection can transport a number of these nuclei into the bulk material and strongly affect the glass-forming ability of the entire system. Under low-gravity conditions (1) the majority of the nuclei should remain at the container wall and (2) the homogeneity and microstructure of the solidified glasses should differ significantly from those produced on Earth. Therefore, the glasses and glass-ceramic materials processed under low-gravity and Earth conditions should be totally different.

This Spacelab D1 experiment was the eighth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011, TEXUS 11, TEXUS 12). (The series of experiments as a whole included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objectives of this study were to (1) investigate the solidification of glasses in the absence of gravity-induced convective effects and (2) investigate if (a) phase separation is promoted by existing nuclei or (b) phase separated regions can act as nuclei.

Six samples of the Li_2O-SiO_2 system (24.00 to 34.00 mol% Li_2O) and two samples of the Na_2O-B_2O_3-SiO_2 system were contained in a glass-like carbon crucible (see Reference (5) for exact sample compositions). Three of the Li_2O-SiO_2 samples and one of the Na_2O-B_2O_3-SiO_2 samples contained 0.01 mol% Pt. (The Pt was added as a nucleating agent.) The Li_2O-SiO_2 system exhibits phase separation and crystallization and, therefore, may be considered a model for glass-ceramic materials. The Na_2O-B_2O_3-SiO_2 system exhibits phase separation only and, therefore, represents a glass material.

During the Spacelab D1 mission, the Isothermal Heating Facility (IHF) was used to melt and resolidify the samples. Two experimental runs were performed using different cooling rates. Run #1 (designated as WL-IHF 05A) was performed with the following parameters:

heating rate = 88.4 K/min
holding time = 18.1 min
Tmax = 1300 ^{o}C
cooling rate = from 1300 to 970 ^{o}C: 49.8 K/min
from 970 to 350 ^{o}C: 32.4 K/min

Run #2 (designated as WL-IHF 05B) was performed with the following parameters:

heating rate = 87.8 K/min
holding time = 18 min
Tmax = 1290 ^{o}C
Cooling rate = from 1290 to 970 ^{o}C: 9 K/min
from 970 to 850 ^{o}C: 4.2 K/min
from 850 to 350 ^{o}C: 26.4 K/min

<Note: It was not clear which samples were processed during each run.>

"It should be mentioned that in all cases an [undesired] temperature gradient of... [about]... 13 K/cm parallel to the cartridge axis was observed, even during the holding period at 1300 ^{o}C." (5, p. 167) <Note: The Experiment Investigator reported that this thermal gradient did not adversely affect the results from the experiment.> Corresponding samples were processed on Earth for comparison.

Post-flight examination of the samples included electron microprobe analysis (Christiansen-Shelyubskii method) and scanning electron microscopy (SEM). It was reported that (compared to the 1-g samples) the low-gravity processed materials contained a nearly homogeneous composition distribution. In all the flight

samples, any inhomogeneous regions were in the "...contact zone... wall-melt." (5, p. 168) The addition of Pt to the low-gravity samples produced in a more uniform and fine-grained structure. <Note: Although not specifically stated in Reference (5), it appears these results apply only to the Li_2-SiO_2 samples.>

The crystalline phase Li_2 . 2 SiO_2 in the Li_2-SiO_2 system illustrated a sperulitic growth under 1-g conditions. However, under low-gravity conditions, this phase was dendritic. <Note: The reason for this difference had not yet been determined at the time available publications were written.> In the Na_2O-B_2O_3-SiO_2 system, both low-gravity and 1-g processed samples illustrated a type of microstructure formed by a spinodal-type phase separation process.

No further information concerning this experiment could be located at this time.

Key Words: Diffusion, Glasses, Glass Melts, Glass Formation, Ceramics, Binary Systems, Model Materials, Melt and Solidification, Separation of Components, Phase Separation, Nucleation, Homogeneous Nucleation, Heterogeneous Nucleation, Nucleating Agents, Mass Transfer, Reaction Kinetics, Thermal Soak, Thermal Gradient, Composition Distribution, Spinodal Decomposition, Cooling Rate, Solid/Liquid Interface, Dendritic Structure, Buoyancy-Driven Convection, Crystal Homogeneity, Crystalline Structure, Grain Structure, Crucible Effects, Thermal Environment More Extreme Than Predicted

Number of Samples: eight
Sample Materials: Six samples of the Li_2O-SiO_2 system (24.00 to 34.00 mol% Li_2O) and two samples of the Na_2O-B_2O_3-SiO_2 system. Three of the Li_2O samples and one of the Na_2O samples contained 0.01 mol% Pt
(Pt*, Li*O*Si*O*, Na*O*B*O*Si*O*)
Container Materials: glass-like carbon
(C*)

Experiment/Material Applications:
Both the Li_2O-SiO_2 and the Na_2O-B_2O_3-SiO_2 systems are glass formers. "Li_2O-SiO_2 shows phase-separation and crystallization under certain conditions and may be regarded as a model system for glass-ceramic materials. Na_2O-B_2-SiO_2 displays phase-separation only and represents materials of the Vycor-type porous glass and of pyrex-type glass." (5, p. 167)

References/Applicable Publications:
(1) Braetsch, V. and Frischat, G. H.: Homogeneity of Li_2O-SiO_2 Glasses as Prepared Under Microgravity and 1-g Conditions. Natur wissenschaften, 73, Jahrgang Heft 7, July 1986, pp. 368-369.

(2) Input received from Experiment Investigator, August 1988 and August 1993.

(3) Braetsch, V. and Frischat, G. H.: Homogeneity of Glasses as Prepared Under Microgravity and 1-g Melting Conditions. In BMFT/DFVLR Scientific Results of the German Spacelab D1 Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 45. (abstract only)

(4) Frischat, G. H. and Braetsch, V.: Homogeneity of Glasses. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 75-76. (preflight)

(5) Braetsch, V. and Frischat, G. H.: Homogeneity of Glasses as Prepared Under Microgravity and 1-g Melting Conditions. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 166-171. (post-flight)

(6) Braetsch, V. and Frischat, G. H.: Influence of Microgravity on the Homogeneity of Glasses. Proceedings of the 6th European Symposium on Materials Sciences Under Microgravity Conditions, Bordeaux, 1986, pp. 259-262.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 13
Launch Date/Expt. Date: April 1986
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1: isothermal four-chamber furnace. (Furnaces from the TEM 01-1 module have been used several times on the TEXUS flights.)
Builder of Processing Facility: ERNO Raumfahrttechnik GmbH, Federal Republic of Germany

Experiment:
Reaction Kinetics in Glass Melts

This TEXUS 13 experiment was the ninth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011, TEXUS 11, TEXUS 12, Spacelab D1). (The series of experiments, as a whole, included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objective of this experiment was to obtain data which, when combined with data from earlier studies, would (1) enable the formation of possible mathematical models describing the diffusive transport of migrating species during glass solidification and (2) "...supply information about the occurring reciprocal effects of the migrating species during diffusive transport." (1, p. 27, translation) <Note: The specific reciprocal effects were not detailed in the available publications.>

Two samples were processed during the mission each comprised of cylindrical glass materials. The interdiffusion sample consisted of one cylinder of Na_2O . 3 SiO_2 and one cylinder of Rb_2O . 3 SiO_2. The self diffusion sample consisted of two cylinders of 0.5 Na_2O .0.5 Rb_2O . 3 SiO_2 with a layer of Na^{22} sandwiched in between. The glass samples were encased in Pt and placed in a Ni cartridge. <Note: The Principal Investigator reported that samples were configured similarly as described in Frischat, TEXUS 11.>

During the TEXUS 13 mission, one chamber of the TEXUS Experiment Module TEM 01-1, Isothermal, Four-Chamber Furnace was used to process the glass cylinders. The material was heated to 1425 K. An effective low-gravity experimentation time of 250 seconds was

reported.

Post-flight examination of the interdiffusion section of the sample was accomplished using a microprobe while that of the tracer section of the sample was accomplished using residual activity methods. The following self diffusion coefficient, D^*_{Na}, and interdiffusion coefficient, $D^{\sim}_{Na,Rb}$, were reported:

For x = 0.5 mol.% and T = 1426 K:
$D^*_{Na} = (1.04 +/-0.12) \times 10^{-5} cm^2/s$
$D^{\sim}_{Na,Rb} = (8 +/-4) \times 10^{-6} cm^2/s$

The value for D^*_{Na} fits very well with the data obtained from earlier low-gravity studies. The value for $D^{\sim}_{Na,Rb}$ confirmed the data obtained from the TEXUS 1 and Spacelab 1 experiments (see Frischat, TEXUS 1 and Spacelab 1 (this chapter)). No concentration dependence on $D^{\sim}_{Na,Rb}$, which was present at temperatures below 1300 K, was observed. "The validity of one of the theoretical approaches... [known] so far--which establish[ed] a relationship between self-diffusion and interdiffusion coefficients--however could not be verified because accurate D^*_{Rb} values were missing." (1, p. 29, translation).

<Note: It is not clear to the editors why it was not possible to obtain accurate D^*_{Rb} values.>

No further information concerning the results of this experiment could be located at this time.

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Binary Systems, Model Materials, Melt and Solidification, Diffusive Mass Transfer, Diffusion Coefficient, Interdiffusion, Liquid/Liquid Diffusion, Double Diffusion, Self-Diffusion, Tracer Particles, Buoyancy-Driven Convection, Concentration Distribution, Reaction Kinetics, Solid/Liquid Interface, Liquid/Liquid Interface, Optics Applications

Number of Samples: Two: an interdiffusion section and and self-diffusion section (see **Experiment** section (above))
Sample Materials: $xNa_2O-(1-x)Rb_2O-SiO_2$, X = 0.5, alkali silicate melts
(Na*O*Rb*O*Si*O*)

Container Materials: Glass sample encased in platinum and then enclosed in a nickel cartridge
(Pt*, Ni*)

Experiment/Material Applications:
xNa_2O . $(1-x)Rb_2O$. 3 SiO_2 was used as a model glass system.

See Frischat, TEXUS 1, **Experiment** section (this chapter).

References/Applicable Publications:
(1) Frischat, G. H., Braetsch, V., and Meier, M.: Reaktionskinetik in Glasschmelzen Selbst-und Interdiffusion in Alkalisilicatschmelzen. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 27-29. (post-flight; in German)

(2) Zarzycki, J., Frischat, G. H., and Herlach, D. M.: Glasses. In Fluid Science and Materials Science in Space, Edited by H. U. Walter, Berlin, Heidelberg, New York: Springer, 1987, pp. 549-636.

(3) Input received from Principal Investigator, August 1988 and August 1993.

(4) Meier, M., Braetsch, V., and Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. In Journal of the American Ceramic Society, Vol. 73, Number 7, 1990, pp. 2122-2123. (post-flight; partial results from TEXUS 11, TEXUS 12, TEXUS 13, and TEXUS 20 experiments)

(5) Self- and Interdiffusion in Alkali-Silicate Melts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 130-131. (post-flight)

(6) Frischat, G. H., Braetsch, V., and Meier, M.: Reaktionskinetik in Glasschmelzen - Selbst-und Interdiffusion in Alkalisilicatschmelzen. TEXUS 13, BMFT Research Report 01-QV-334 A5, October 1986.

(7) Meier, M., Braetsch, V., Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. J. Am. Ceram. Soc., 73 (7), 1990, pp. 2122-2123.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 15
Launch Date/Expt. Date: May 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Reaction Kinetics in Glass Melts

This TEXUS 15 sounding rocket experiment was the tenth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011, TEXUS 11, TEXUS 12, Spacelab D1, TEXUS 13). (The series of experiments as a whole, included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.) The specific objective of this experiment was to study the self-diffusion in two glass melts with the compositions: (1) 0.75 Na_2O . 0.25 Rb_2 . 3 SiO_2 and (2) 0.5 Na_2O . 0.5 Rb_2 . 3 SiO_2.

It was reported that shortly after the successful launch of the rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burnthrough of the second stage of the rocket had occurred and the stage, in turn, had collided with the prematurely separated payload. The upper part of the payload, including the TEXUS TEM 01-1 experiment module, parachuted to the Earth undamaged.

The Experiment Investigator reported that there were no results from this experiment.

The experiment was first reflown on TEXUS 16 (see Frischat, TEXUS 16 "Self-Diffusion") and then (successfully) on TEXUS 20.

<Note: The experimental results from TEXUS 20 are beyond the scope of this Technical Memorandum (TM). These results will be reported in later versions of this TM.>

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Model Materials, Melt and Solidification, Self-Diffusion, Liquid/Liquid Diffusion, Reaction Kinetics, Solid/Liquid Interface, Payload Survivability, Acceleration Effects, Rocket Failure

Number of Samples: two
Sample Materials: 0.75 Na_2O . 0.25 Rb_2 . 3 SiO_2 and 0.5 Na_2O. 0.5 Rb_2 . 3 SiO_2
(Na*O*Rb*O*Si*O*)
Container Materials: platinum
(Pt*)

Experiment/Material Applications:
Alkaline silicate glass melts are models for technological glasses.

See Frischat, TEXUS 1, **Experiment** section (this chapter).

References/Applicable Publications:
(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 107-108. (in German; post-flight)

(2) Meier, M., Braetsch, V., Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. J. Am. Ceram. Soc., 73(7), 1990, pp. 2122-2133.

(3) Input received from Experiment Investigator, August 1993.

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-1
Builder of Processing Facility: Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

Experiment:
Self-Diffusion

This TEXUS 16 sounding rocket experiment was the eleventh in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011, TEXUS 11, TEXUS 12, Spacelab D1, TEXUS 13, TEXUS 15). (The series of experiments as a whole included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion of glass melts.) The specific objective of this experiment was to study the self-diffusion in alkaline silicate glass melts. Reportedly, two samples (1) 0.75 Na_2O . 0.25 Rb_2 . 3 SiO_2 and (2) 0.5 Na_2O . 0.5 Rb_2 . 3 SiO_2 were to be heated to 1525 K and cooled down.

It was reported that shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all the experiment modules including the TEM 01-1 module.

The Experiment Investigator reported that there were no results from this experiment.

The experiment was successfully reflown on TEXUS 20.

<Note: The experimental results from TEXUS 20 are beyond the scope of this Technical Memorandum (TM). These results will be reported in later versions of this TM.>

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Melt and Solidification, Self-Diffusion, Liquid/Liquid Diffusion, Reaction Kinetics, Solid/Liquid Interface, Payload Survivability, Acceleration Effects, Rocket Failure, Payload Recovery System Failure

Number of Samples: two
Sample Materials: 0.75 Na_2O . 0.25 Rb_2 . 3 SiO_2 and 0.5 Na_2O . 0.5 Rb_2 . 3 SiO_2
(Na*O*Rb*O*Si*O*)
Container Materials: Platinum
(Pt*)

Experiment/Material Applications:
See Frischat TEXUS 1, 6, 11, 12, 13, 15 (this chapter).

References/Applicable Publications:
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 109-111. (in German; post-flight)

(2) Meier, M., Braetsch, V., Frischat, G. H.: Self Diffusion in Na_2O-Rb_2O-SiO_2 Glass Melts as Obtained by Microgravity Experiments. J. Am. Ceram. Soc., 73(7), 1990, pp. 2122-2133.

(3) Input received from Experiment Investigator, August 1993.

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Co-Investigator(s): None
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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: MBB/ERNO, Bremen, Germany

Experiment:
Refraction Gradients in Glasses

This TEXUS 16 sounding rocket experiment was the twelfth in a series of investigations designed by Frischat et al. to study reaction kinetics in glasses (see Frischat, TEXUS 1, TEXUS 4, TEXUS 6, Spacelab 1, STS-011, TEXUS 11, TEXUS 12, Spacelab D1, TEXUS 13, TEXUS 15, TEXUS 16("Self-diffusion")). (Earlier experiments in the series of experiments included examinations in the areas of (1) interdiffusion between glass melts of differing compositions, (2) corrosion of silica by the melt, and (3) self-diffusion in glass melts.)

Reportedly, the specific objective of the experiment was to study a "Silver/sodium exchange... at 350 ^{o}C between an alumo-alkali-silicate glass and a... silver containing salt melt." (Reference 3) <Note: No other details concerning the experimental objectives could be located at this time.>

Two samples contained within one cartridge were to be processed during the mission: (1) 61 SiO_2 . 33 Na_2O . 6 Al_2O_3 (glass) and 9 $NaNO_3$. 1 $AgNO_3$ (salt melt). <Note: No other details describing the expected inflight procedures could be located at this time.>

It was reported that shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including the TEM 01-2 module.

The Experiment Investigator reported that there were no experimental results. The experiment was reflown on TEXUS 19. <Note: The results from the TEXUS 19 mission are beyond the scope of this Technical Memorandum (TM). These results will be published in later versions of this TM.>

Key Words: Diffusion, Glasses, Glass Melts, Alkaline Glasses, Molten Salts, Melt and Solidification, Interdiffusion, Double Diffusion, Liquid/Liquid Diffusion, Reaction Kinetics, Ion Exchange, Solid/Liquid Interface, Liquid/Liquid Interface, Payload Survivability, Acceleration Effects, Rocket Failure, Payload Recovery System Failure

Number of Samples: two
Sample Materials: (1) Glass: 61 SiO_2 . 33 Na_2O . 6 Al_2O_3
(2) Salt Melt: 9 $NaNO_3$. 1 $AgNO_3$
(Si*O*Na*O*Al*O*, Na*N*O*Ag*N*O*)
Container Materials: platinum
(Pt*)

Experiment/Material Applications:
The experiment was performed to investigate if "...puried silver profiles can be obtained by a one step process with an unstirred salt melt." (Reference (3)) <Note: The meaning of "puried silver profiles" was not clear to the editors.>

References/Applicable Publications:
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 109-111. (in German; post-flight)

(2) Frischat, G. H. Horst, U., and Beier, W.: Special Refractive Index Profiles in Glasses Obtained by Ion Exchange. TEXUS 20, BMFT Research Report 01 QV 86201, 1989. (in German)

(3) Input received from Experiment Investigator, August 1993.

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Experiment Origin: Federal Republic of Germany
Mission: TEXUS 8
Launch Date/Expt. Date: May 1983
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 06-8: Flowing junction cell equipped with shadow optics
Builder of Processing Facility: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Diffusion Boundary

This TEXUS 8 experiment was the first in a series of investigations designed by Merkens et al. to study the interdiffusion of molten salts. The specific objective of the experiment was to bring two columns of molten salts into contact and observe the subsequent diffusion boundary.

The experiment employed a special type of diffusion cell called a flowing junction cell. Because this cell-type (1) had never been employed in the reduced gravity environment and (2) was to be employed during the experiments of Merkens et al. on the (later) Spacelab D1 mission (see Merkens, D1 (this chapter)), a secondary objective of the experiment was to test the ventilation and filling capabilities of the cell. (Such capabilities are essential for the establishment of a diffusion boundary in the cell.)

During the mission, melts of two different compositions were fed into the stainless steel diffusion cell from two compressible reservoirs. <Note: It appears that one of these melts was a mixture of sodium nitrate and silver nitrate with a mole fraction of $AgNO_3$3 of 0.84, and that the other of the melts was a mixture of sodium nitrate and silver nitrate with a mole fraction of $AgNO_3$ of 0.86.> Synchronized injection of the melts pressed the air out of side slits in the cell of ca. 0.12 mm thickness. The cell was equipped with quartz-glass windows to permit observation of the subsequent diffusion. Poor wetting of the melts was insured by siliconization of the walls and windows. Photographs of the diffusion boundary were obtained (via a shadow optic system) and recorded on 16 mm film.

Reportedly, "The melts flowed from both sides of the cell towards each other and came into contact where the interface was to be located." (6, p. 138) After ventilation, a diffusion boundary of 0.12 thickness formed. This high quality, plane interface was free of gas inclusion and precise diffusion measurements could be performed.

No further information (published in English) concerning these measurements could be located at this time. <Note: References (3), (4), and (7) could not be located prior to the preparation of this summary.>

Key Words: Diffusion, Molten Salts, Binary Systems, Interdiffusion, Double Diffusion, Liquid/Liquid Diffusion, Diffusion Boundary, Diffusive Mass Transfer, Wetting, Non-Wetting of Container, Surface Tension, Flowing-Junction Cell, Liquid Reservoir, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Ventilation, Interface Shapes, Concentration Distribution, Inclusions, Interface Physics, Shadow Optics

Number of Samples: one
Sample Materials: Silver nitrate and sodium nitrate melts with mole fractions of silver nitrate of 0.84 and 0.86.
(Ag*N*O*, Na*N*O*)
Container Materials: Stainless steel cell with quartz glass windows (dimensions of the cell: 40 X 10 X 5 mm^3).
(Si*O*)

Experiment/Material Applications:
This experiment examined the ventilation and filling of controlled-wetting thermodiffusion cells under low-gravity.

References/Applicable Publications:
(1) Richter, J. and Hahne, A.: Thermodynamics of Irreversible Processes in Molten Salts: Problems of Interdiffusion Measurements Under Microgravity. In ESA 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-7, 1983, ESA SP-191, pp. 153-157. (no TEXUS results mentioned specifically)

(2) Gobeler, E., Hermanns, J., Merkens, W., and Richter, J.: Diffusion Measurements in Molten Salts Under 1-g and Low-Gravity Conditions. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 163-164. (specifically, post-flight)

(3) Hahne, A., Hermanns, J., and Richter, J.: Measurement of the Concentration Dependence of Interdiffusion Coefficients in Fused Salts. Z. Flugwiss. Weltraumforschung, 8, p. 334, 1982.

(4) Merkens, W., Hermanns, J., and Richter, J.: Filling and Ventilation of a Diffusion Cell Under Reduced Gravity. Z. Flugwiss. Weltraumforschung, 8, p. 415, 1984.

(5) Input received from Principal Investigator R. Richter, August 1989.

(6) Richter, J., Hermanns, J., Merkens, W.: Ventilation and Filling of an Optical Diffusion Cell. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 138-139. (post-flight)

(7) Richter, J., Hermanns, J., Merkens, W., and Hahne, A.: Entlüften und Befüllen einer Diffusionszelle-TEXUS 8. Abschlussbericht, BMFT-1984. (in German)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Process Chamber Double Rack
Processing Facility: Flowing-junction cell (previously tested on TEXUS 8). Experiment setup included Savart interferometry.
Builder of Processing Facility: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

Experiment:
Interdiffusion of Molten Salts (PK-IDS 00)

The measurement of differential interdiffusion coefficients is of common interest to theoretical and experimental researchers. In the case of molten salts, the precise measurement of such coefficients on Earth is very difficult and often impossible. At high temperatures, the presence of gravity-driven convection affects the interdiffusion region and, thus, the measurements.

"To perform precise measurements of diffusion coefficients, it is necessary to generate the sharpest possible concentration step between the molten salts of different composition. The solution of Fick's differential equation used is based on a constant diffusion coefficient within the concentration range considered. This means that...[diffusion] coefficients should be measured with the smallest possible concentration differences. Of all experiments for the determination of the concentration dependent diffusion coefficients, the optical methods meet this requirement best." (2, p. 73)

This experiment was the second in a series of investigations designed by Merkens et al. to study the interdiffusion of molten salts (see Merkens, TEXUS 8). The specific objective of the experiment was to determine the inter-diffusion coefficient of a binary $NaNO_3$ + $AgNO_3$ melt. (Reportedly, the melt had a mole fraction of silver nitrate of 0.85 and a concentration difference of 0.02.)

Ventilation and filling of the diffusion cell (called a flowing junction cell) had been tested previously on TEXUS 8 (see Merkens, TEXUS 8). The similar STS-030 cell consisted of two

compressible reservoirs from which melts of the two different compositions were fed into the diffusion cell. <Note: The exact compositions of these melts were unclear to the editors.> Synchronized injection of the melts forced air out of side slits in the cell. Optical analysis of the cell was made possible by employing a Savart interferometer.

In order to achieve homogeneous melts in the reservoirs, the cell was heated for 24 hours at a temperature of 300 deg ^{o}C. Reportedly, because of delays during the ventilation and filling procedure (jamming of the slide bars associated with the compressible reservoirs), the applied anti-wetting coating was destroyed and the necessary phase boundary could not be formed. Thus, no diffusion measurements were possible.

No further information concerning this experiment could be located at this time.

Key Words: Diffusion, Molten Salts, Binary Systems, Interdiffusion, Double Diffusion, Liquid/Liquid Diffusion, Diffusion Boundary, Diffusion Coefficient, Fick's Law, Diffusive Mass Transfer, Wetting, Non-Wetting of Container, Coated Surfaces, Surface Tension, Flowing-Junction Cell, Concentration Distribution, Solutal Gradients, Buoyancy-Driven Convection, Liquid Reservoir, Liquid Injection, Liquid Transfer, Liquid/Liquid Interface, Interface Physics, Ventilation, Interferometric Measurement, Hardware Malfunction

Number of Samples: One flowing junction cell.
Sample Materials: Silver nitrate and sodium nitrate melt (The melt had a mole fraction of silver nitrate of 0.85 and a concentration difference of 0.02.)
(Na*N*O*, Ag*N*O*)
Container Materials: Unknown (May have been the same as that employed on TEXUS 8: stainless steel cell with quartz glass windows.) The anti-wetting coating was not specified.
(Si*O*)

Experiment/Material Applications:
Molten salt transport properties such as diffusion coefficients, ultrasonic absorption coefficients, thermal conductivity, etc. are difficult to obtain. For example, diffusion coefficient

measurements are hindered because gravity-induced convection occurs in the sharp concentration gradient between molten salts of different compositions. In these low-gravity investigations, diffusion measurements were expected to be made with reduced distortion from this convection.

Technical processes controlled by diffusion require measurements of such transport properties. These technologies include melt electrolysis of aluminum and finishing of metals and glasses.

References/Applicable Publications:

(1) Richter, J. and Merkens, W.: Interdiffusion in Molten Salts. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 91. (abstract only; post-flight)

(2) Merkens, W. and Richter, J.: Interdiffusion in Molten Salts. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 73. (preflight)

(3) Richter, J., Hermanns, J., and Merkens, W.: Ventilation and Filling of a Diffusion Cell Under Reduced Gravity. Zeitschrift für Flugwissenschaften und Weltraumforschung, Vol. 8, November-December 1984, pp. 415-419. (in German; preflight)

(4) Gobeler, E., Hermanns, J., Merkens, W., and Richter, J.: Diffusion Measurements in Molten Salts Under 1-g and Low-Gravity Conditions. Proceedings of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 160-165.

(5) Richter, J. and Hahne, A.: Thermodynamics of Irreversible Processes in Molten Salts: Problems of Interdiffusion Measurements Under Microgravity. In proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, pp. 153-160. (preflight)

(6) Hahne, A., Hermanns, J., and Richter, J.: Measurement of the Concentration Dependence of Interdiffusion Coefficients in Fused Salts. Z. Flugwiss. Weltraumforschung, 8, p. 334 (1982).

(7) Merkens, W., Hermanns, J., and Richter, J.: Filling and Ventilation of a Diffusion Cell Under Reduced Gravity. Z. Flugwiss. Weltraumforschung, 8, p. 415, 1984.

(8) Input received from Principal Investigator J. Richter, August 1989.

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Co-Investigator(s): Unknown
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Experiment Origin: France
Mission: STS Launch #9, STS-9 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Rack
Processing Facility: Low Temperature Gradient Heating Facility (GHF) Furnace
Builder of Processing Facility: Unknown

Experiment:
Thermodiffusion in Tin Alloys 1ES320

During a solidification process, a thermal gradient is established within the melt. The gradient can drive the separation (or thermomigration) of components in the system. On Earth, convective flow tends to disturb the migration process, making it difficult to determine the extent of the thermomigration effect.

This Spacelab 1 experiment was the first in a series of investigations designed by Malmejac and/or Praizey et al. to study thermomigration (Soret effect) in liquid alloys under low-gravity conditions. (The experiment was also one of two Spacelab 1 investigations designed by Malmejac et al. to examine the behavior of metallic melts (see Malmejac, Spacelab 1, "Nucleation Behavior of Eutectic Alloys" (Chapter 14)).) The specific objective of this study was to determine thermodiffusion coefficients of tin alloys in a reduced gravity environment.

Prior to the mission, four 2-mm diameter, 18-mm long samples of Sn-0.4 wt.% Co were prepared and placed within zirconia shear cells. During the mission, the cells were placed within a gradient furnace and heated such that a thermal gradient of 200 ^{o}C/cm was established. After a 6-hour soak period (chemical steady state), each sample was divided into six parts (while still molten) by rotating each of six portions within the shear cell a slightly different distance. The samples were then solidified, freezing the Co content in each portion (see References (2) and (3) for shear cell description).

Ground-based samples, processed in a vertical position with cold end down, were used for comparison.

Post-flight, it was reported that twelve sample portions were available for analysis. Of these twelve, three had an initial mean Co concentration "...such that there might have been some other effects than thermomigration." (1, p. 147) Of the nine that were reportedly analyzed, the results from six sample portions were discussed.

Each part was analyzed separately by a neutron activation method. "The accuracy of the concentration measurements is in the range of a few permil[sic]." (1, p. 147) The mean weight concentrations of Co for the six flight sample measurements reported, ranged from 1.385 wt.% at the hot end to 0.689 wt.% at the cold end. The mean values for the 1-g processed samples were given as 1.002 wt.% (hot end) and 0.992 (cold end). Convection calculations, performed using a unidirectional solutal approach and a radial gradient approach, demonstrated "...that convection on earth hides the effect of the thermomigration process as shown by [the] earth based results." (1, p. 151) (See References (1) and (3) for convection calculations.)

The concentration results for the low-gravity experiment allowed calculation of the heat of transport and the Soret coefficient. However, "The accuracy is about 1% for Co concentration measurements but only... +/-... 10 ^{o}C as regards temperature values, that in the absence of real flight data are only estimated from numerous thermal tests." (1, p. 148)

The heat of transport was reported to be +/- 2% accuracy):
$Q(Cal/mole) = -(3.72 +/- 0.008)T(K)$ for $593\ K < T < 933\ K$

The associated Soret coefficient was given as:
$S_T = 1.86/T$

Key Words: Diffusion, Melt and Solidification, Binary Systems, Alloys, Thermal Diffusion, Thermomigration, Diffusion Coefficient, Thermal Gradient, Thermal Soak, Buoyancy-Driven Convection, Concentration Distribution, Soret Effect, Heat of Transport, Heat and Mass Transfer, Solid/Liquid Interface, Shear Cell

Number of Samples: four
Sample Materials: Sn-0.4 wt.% Co
(Sn*Co*)
Container Materials: zirconia
(Zr*)

Experiment/Material Applications:
A tin-cobalt system was selected for study because (1) "...the low melting point of tin allows an important thermal gradient, (2) "...the difference of atomic mass between tin and cobalt leads to an important thermomigration effect, (3) "...the cobalt concentration is easy to analyze accurately by activation methods, (4) "...cobalt has a destabilizing solutal effect on earth, which increases the interest of a microgravity experiment on such a system." (1, p. 147)

The heat of transport, Q, must be determined to solve the general transport phenomena equations. If the transport is determined via low-gravity research, accurate measurements can be assured.

References/Applicable Publications:
(1) Malmejac, Y. and Praizey, J. P.: Thermomigration of Cobalt in Liquid Tin: Experiment 1 ES 320. In ESA 5th European Symposium On Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 147-152.

(2) Praizey, J. P.: Technical Implications of a Flight Experiment Design. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, pp. 127-132 (ESA SP-191). (preflight; discusses experimental apparatus)

(3) Praizey, J. P.: Benefits of Microgravity for Measuring Thermotransport Coefficients in Liquid Metallic Alloys. In International Journal of Heat and Mass Transfer, Vol. 32, No. 12, 1989, pp. 2385-2401. (post-flight; discusses experimental apparatus; Spacelab 1 and Spacelab D1 results)

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
Launch Date/Expt. Date: November 1983
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Spacelab Material Science Double Rack (MSDR)
Processing Facility: High Temperature Thermostat (HTT)
Builder of Processing Facility: Technische Universität Berlin, Institut für Metallforschung, Germany

Experiment:
Self-Diffusion in Liquid Metals (Sn) 1ES335

In a liquid-metal system, diffusion-coefficient temperature dependence is difficult to resolve. Significant discrepancies in experimental measurements of the coefficients, for example, indicate a degree of inaccuracy in diffusion results. Thus, a multitude of theories has been formulated to describe diffusion coefficient dependencies. It is generally accepted, however, that in a molten system, gravity-driven convection (and possibly gravity-independent Marangoni convection) contributes to the resultant diffusion coefficient. In addition, a phenomenon known as the "wall effect" (which is related to the sample capillary diameter) may affect the diffusion behavior near the containing walls.

This Spacelab 1 experiment was the first in a series of investigations designed by Wever et al. to study diffusion in liquid metals. The overall objective of the experiment was to study the self diffusion of Sn^{112} and Sn^{124} in liquid tin. The specific goals of the research were to (1) determine the self diffusion coefficient of molten tin without the disturbing influence of gravity-driven convection, (2) compare the temperature dependence of the diffusion coefficient with theories and terrestrial-experimental results, and (3) determine the extent of the isotope and "wall" effects.

During the mission, 16 tin samples (with Sn^{112} and Sn^{124} tracers) were isothermally annealed within eight vacuum chambers in the Spacelab High Temperature Thermostat (HTT). It appears that each of the eight chambers (1) held two samples (one which had a capillary diameter of 1 mm and one which had a capillary diameter of 3 mm), (2) was heated to a different diffusion temperature (ranging from 240-1250 ^{o}C), and (3) employed three thermocouples (to monitor thermal distributions). <Note: It is not clear if

each of the 16 tin samples contained both Sn^{112} and Sn^{124} tracers, although it appears from discussions of the post-flight sample analysis that both isotopes were in each of the samples. It was reported that the "...two highly enriched stable isotopes... were used as tracers in a disc of about 4 mm thickness at the top of the samples." (4, p. 147)>

The HTT employed two heaters: one to provide a temperature gradient during melting and cooling and one to provide an isothermal distribution during annealing. During the experiment sequence, each chamber was : (1) heated to a temperature above its melting point, (2) heated to its appropriate diffusion temperature, (3) maintained at the diffusion temperature for a specified time (for example, 2.7 hours (400 ^{o}C) and 10.5 hours (260 ^{o}C)), and (4) cooled via the helium flooding.

It appears that difficulties related to (1) unexpected vacuum-gas-system (VGS) operation, (2) experimental data loss, and (3) a HTT power relay problem, severely affected diffusion results in at least two of the chambers. In addition, while precise temperature measurement was of significant importance, errors in all temperature values were higher than anticipated (1% instead of +/- 0.1%).

At the time Reference (1) (below) was written, post-flight concentration profiles of Sn^{112} and Sn^{124} had been made post-flight from 1-mm capillary samples housed in (successful) chambers #2, #5 and #6. These three samples had diffusion temperatures of 810 ^{o}C, 1090 ^{o}C, and 398 ^{o}C.

Reportedly, the concentration profiles of the 810 ^{o}C and 398 ^{o}C samples indicated a high degree of accuracy of the data points (0.5%) when compared to 1-g experimental data. <Note: "A high degree of accuracy of the data points" is not clear to the editor. The reader is referred to Reference (1) to view the data corresponding to the concentration profiles.> However, "The results of the 1090 ^{o}C sample [showed] larger steps in the concentration profile." (1, p. 202) It was suspected that these steps may have been related to (1) Marangoni convection in the liquid system, (2) spacecraft accelerations during the diffusion anneal, or (3) preparation/handling for the post-flight analysis.

<Note: Reference (4), which was published after Reference (1), clearly presented results of Spacelab 1 samples with diffusion temperatures of 321 ^{o}C and 850 ^{o}C. This indicates that the eventual sample analysis was not limited to the three diffusion temperatures cited above.> Based on the results reported in References (1) and (4), the following was reported:

(1) The diffusion coefficients for each isotope at 398 ^{o}C and 810 ^{o}C were significantly lower than those determined for 1-g conditions. This decrease indicated a lack of gravity-driven microconvections. These results also implied that 30% to 50% the 1-g diffusion coefficient value was due to gravity-driven convection.

(2) Accuracy of the space-obtained diffusion coefficients were typically "0.5 percent," while accuracy of the Earth-obtained diffusion coefficients had typically been 5%-30%. <Note: It is not clear what an accuracy of 0.5% or 5%-30% percent means.>

(3) "Because of the very good accuracy of the μg-results, it was possible to detect a difference between the diffusion coefficients D_{112} and D_{124} of the different isotope masses 112 and 124...." (4, p. 148) This difference was illustrated in Reference (4) using composition profiles of Sn^{112} and Sn^{124} for the 321 ^{o}C sample (see Reference (4), Figure 8). It appears that from such values, the isotope effect was derived for at least 5 of the samples. A graph depicting the effect illustrated that the isotope effect in Sn generally increased with increasing temperature. Reportedly this fact could be interpreted "...by a more collective atomic motion of atoms at lower temperatures (small free volume) with the tendency to move more gas-like at higher temperatures." (4, p. 148) A discussion of the isotope effect can be found in Reference (4), pp. 146 and 148.>

(4) Further, "...the first sufficient reliable measurements of... [the isotope effect] in liquid diffusion..." were made and were found to be 0.1 (400 ^{o}C) and 0.9 (810 ^{o}C). (1, p. 201) <Note: This statement, which was taken from Reference (1), does not appear to match the isotope effect data presented in Reference (4).>

(5) "The temperature dependence of the μg-diffusion coefficient is not in accordance with the Arrhenius-law.... [Self diffusion coefficient data vs. temperature indicated that (within 2%) the diffusion coefficient is directly proportional to the square of the temperature: $D = K*T^2$ with $K=7.45 * 10^{-11}$ cm^2/K^2s.] The value of the constant K... [was] given for the mean isotope mass m = 118.7 of Sn. The result strongly favors Swalin's fluctuation theory of liquid diffusion. If this model is truly the diffusion mechanism, then the diffusion in liquids is a small step process where no thermal activation is necessary." (4, pp. 147-148) <Note: further details of Swalin's theory is presented in Reference (4).>

(6) "Within the accuracy of the D-values no influence of the capillary diameter could be detected i.e. the wall effect, if present, will be confined to layers of less than 20 microns

thickness near the wall." (4, p. 148)

Key Words: Diffusion, Binary Systems, Self-Diffusion, Diffusion Coefficient, Tracer Particles, Isotope Effect, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Marangoni Convection, Thermal Gradient, Thermal Soak, Solid/Liquid Interface, Wall Effect, Crucible Effects, Coated Surfaces, Annealing, Quench Process, Concentration Distribution, Isotopes, Capillaries, Vacuum, Acceleration Effects, Hardware Malfunction, Deterioration of Samples After Low-G Flight

Number of Samples: sixteen
Sample Materials: Tin doped with Sn isotopes, Sn(112), and Sn(124)
(Sn*)
Container Materials: graphite
(C*)

Experiment/Material Applications:
In order to understand the effect of temperature on the diffusion coefficient and, therefore, to provide a more realistic analysis of the molten liquid system, it is necessary to discern the various diffusion contributions and determine their effects on the overall diffusion mechanism in liquids. Such an understanding is important in solidification processes.

Tin was chosen for this experiment because of its low melting and high boiling temperatures which provide a suitably large temperature range for diffusion studies. Tin has also been widely studied during 1-g self-diffusion experiments, and there exists a large data base for comparison. Tin has many isotopes with a mass difference of up to 10% which makes it well suited for isotope effect studies.

References/Applicable Publications:
(1) Frohberg, G., Kraatz, K. H., and Wever, H.: Self Diffusion Of Sn^{112} and Sn^{124} In Liquid Tin. In ESA 5th European Symposium on Material Science Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 201-205. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements oon Spacelab 1)

(3) Kraatz, K. H.: Interdiffusion in Liquid Metals. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 67-68. (preflight)

(4) Frohberg, G., Kraatz, K. H., and Wever, H.: Atomic Diffusion and Transport in Liquids. In Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 144-151. (Spacelab 1 and D1 data)

(5) Input received from Principal Investigator H. Wever, October 1989.

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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: High Temperature Thermostat (HTT) (Refurbished from Spacelab 1 Mission)
Builder of Processing Facility: Technische Universität Berlin, Institut für Metallforschung, Germany

Experiment:
Self and Interdiffusion in Liquid Metals (Sn-In) (WL-HTT-00)

This experiment was the second in a series of investigations designed by Wever et al. to study the diffusion in liquid metals (see Wever, Spacelab 1). The objectives of this research were similar to those of Spacelab 1, but also included the investigation of (1) the self- and interdiffusion of Sn-In alloys and (2) the extent of the Kirkendall effect.

The Principal Investigator reported that during the mission, 16 Sn/In samples were successfully annealed within the High Temperature Thermostat (HTT). <Note: The actual processing procedure of the experiment was unclear to the editors. The word "annealed" (which appeared in several of the applicable references), used in conjunction with the words "Kirkendall effect" would indicate that the samples were not melted, but were instead annealed, and diffusion across a solid/solid interface was examined. However, from the processing temperatures cited below, it appears that the samples were melted.> A post-flight document (Reference (1)) indicated that the diffusion temperatures were between 260 ^{o}C and 900 ^{o}C. In further reviewing Reference (1), however, it was difficult to discern (1) the exact sample-composition/diffusion-temperature combinations and (2) details concerning the experiment sequence.

It appears that in four of the eight available experiment chambers, samples had one of two alloy combinations. In the first combination, it appears that a section of 85/15 at% In/Sn was separated from a section of 75/25 at% In/Sn by a section of 80/20 at% In/Sn. The 80/20 at% In/Sn was a tracer alloy enriched with natural isotopes Sn^{124} and In^{113}. In the second combination, it

appears that a section of 70/30 at% In/Sn was separated from a section of 60/40 at% In/Sn by a section of 65/35 at% In/Sn. The 65/35 at% sample was a tracer alloy enriched with natural isotopes Sn^{124} and In^{113}. It appears that in the fifth chamber, ("...in order to check the boundary conditions planned at 660 °C[)] one cartridge holds two samples with the same mean composition between them. By comparing the concentration profiles of the two isotopes for regular and blind tests it will be possible to check whether the boundary conditions have been correct or disturbed by Marangoni convection." (4, p. 68)

Because three chambers experienced difficulties during the earlier Spacelab 1 mission (see Wever, Spacelab 1 (this chapter)), it appears that in the remaining three chambers, these Spacelab 1 experiments were repeated (on D1) "without altering experimental data or boundary conditions." (4, p. 68)

At the time Reference (1) was written, post-flight analyses of all the samples had not yet been completed. However, it was reported that:

(1) Low-gravity interdiffusion concentration profiles indicated a high degree of accuracy of the data points (0.7%) when compared to ground-based experimental data. <Note: The meaning of "a high degree of accuracy of the data points" is unclear to the editor. The reader is referred to Reference (1) to view the concentration profile data presentation.>

(2) "As expected the... [low-gravity] diffusion coefficients are lower than the available 1 g-values." (1, p. 149)

(3) "The most surprising result is the temperature dependence of the interdiffusion coefficient which again, as for the self-diffusion of Sn,... [has] a pure square dependence on temperature." (1, p. 149)

It was also reported that no evaluation of the Kirkendall effect could be made.

Key Words: Diffusion, Binary Systems, Metals and Alloys, Self-Diffusion, Interdiffusion, Diffusion Coefficient, Tracer Material, Isotope Effect, Buoyancy-Driven Convection, Marangoni Convection, Kirkendall-Effect, Annealing, Concentration Distribution

Number of Samples: sixteen
Sample Materials: Tin/indium alloy combinations (see experimental writeup for Sn/In combinations)
(Sn*In*)
Container Materials: unspecified, possibly graphite
(C*)

Experiment/Material Applications:
A more accurate determination of diffusion coefficients, as well as such influences as the wall effect, isotope effect, and Kirkendall effect, could be applied to a number of processes in which diffusion (in the liquid state) has an important role. Such processes include (1) homogenization, (2) solidification, (3) crystallization, (4) Ostwald ripening in immiscible alloys, (5) material exchange through boundaries such as in evaporation, (6) phase separation in monotectic systems, and (7) chemical reactions between different phases.

Also see Wever, Spacelab 1 (this chapter).

References/Applicable Publications:
(1) Frohberg, G., Kraatz, K. H., and Wever, H.: Atomic Diffusion and Transport in Liquids. In Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 144-151. (post-flight)

(2) Frohberg, G.: Diffusion and Atomic Transport. In Material Science in Space, Chapter 5, Edited by Feuerbacher, et al., Springer Verlag, Berlin, 1986.

(3) Malmejac, Y. and Frohberg, G.: Mass Transport by Diffusion. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer Verlag, Berlin, 1967.

(4) Kraatz, K. H.: Interdiffusion in Liquid Metals. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 67-68. (preflight)

(5) Frohberg, G., Kraatz, K. H., Wever, H., Lodding, A., and Odelius, H.: Diffusion in Liquid Alloys Under Microgravity. Paper A09 at the DIMETA-Conference, September 1988, Hungary, accepted for publication in the Conference Proceedings. <Note: The current status of this document is unclear.>

(6) Hammacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Sixth European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 413-420. (acceleration measurements on D1)

(7) Frohberg, G., Kraatz, K. H., and Wever, H.: Microgravity Experiments on Liquid- Self and Interdiffusion (Sn, SnIn/Exp. W1-HHT 00.) In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 38. (post-flight)

(8) Frohberg, G., Kraatz, K. H., and Wever, H.: Diffusion and Transport Phenomena in Liquids Under Microgravity. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987, pp. 585-591. (post-flight)

(9) Input received from Principal Investigator H. Wever, October 1989.

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Co-Investigator(s): None
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Experiment Origin: France
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Two separate experiments were performed in the Gradient Heating Facility (GHF) Furnace. The facility was capable of housing three thermocells at one time, thus a total of six thermocells were examined during the mission.
Builder of Processing Facility: C.N.E.S., France

Experiment:
Measurement of the Thermally Induced Ludwig-Soret Effect (WL-GHF 01)

"When a temperature gradient is maintained through a binary solution, a diffusion phenomenon appears, which leads to the separation of the constituents.... [This phenomenon] is called the Ludwig Soret effect." (5, p. 69) Thermal diffusion experiments, which seek information about the Soret coefficient and system transport phenomena, are often hindered by gravity-induced flow instabilities.

The objective of this Spacelab D1 experiment was to observe the thermal diffusion of a molten mixture, "...in such conditions that the gravity field is small compared to the thermal field, and permitting in-situ measurement of the Soret effect after a careful choice of the method and dimensions of the cell." (11, p. 152)

Although it appears that only one experiment run was planned, a second experimental opportunity seems to have been made available at the end of the mission after the processing facility had been used for all other scheduled experiments. The two runs were essentially the same except that during the second, the D1 vestibular sled facility, "...consisting of a motor driven seat and a 4 m runway which is fixed in the Spacelab aisle...", (10, p. 53) was operating.

During the two investigations, a total of six thermodiffusion cells containing a molten salt AgI/KI mixture were heated within the Gradient Heating Facility (GHF). A strong temperature

gradient of 70 ^{o}C/cm (resulting in an overall temperature difference of 200 ^{o}C-240 ^{o}C) was imposed over the cells for 6 hours. As expected, there was a salt volume increase during melting. This increase was absorbed with a porous material cylinder which surrounded the salt. Detection of the diffusion in real time was achieved by measuring the thermoelectric power with silver electrodes. During the melting, therefore, it was important to compensate for the volume expansion while keeping the cell completely filled and fluid in contact with the electrodes.

Space diffusion experiments were compared to similarly performed Earth diffusion experiments. Reportedly, a few hours after Earth-based experiment initiation, thermoelectric signals were erratically perturbed. It was thought that these perturbations were due to liquid contact problems in the top electrode. Too large of a liquid absorption by the porous tube near the top electrode had occurred. (Such perturbations did not appear in the space samples.) This proved that in space the measured thermopotential corresponded to a complete cell filling.

The evolution of the space measured thermoelectric power with time was quite different from equivalent Earth variations. It seemed characteristic of a diffusion process but slower than predicted, corresponding to much smaller diffusion coefficients in space than on Earth (about 5 times smaller). The Soret coefficient could not be calculated, as the stationary state was too far from being attained, but it is positive when related to the migration of AgI toward the cold side of the cell.

Small Earth variations make it impossible to measure the Soret effect correctly under normal gravity conditions. Although the system was theoretically stable, the heavier components migrate toward the cold side of the cell.

Resultant vestibular sled accelerations were correlated to experimental measurements during the second experimental run. It was found that accelerations produced by the sled were at least 10 times smaller than constant accelerations measured in the Materials Science Double Rack. In addition, a numerical simulation of the convection in the case of the long, differentially heated cylinders corresponding to the space situation confirmed that the experiment was not perturbed by convection resulting from the low-gravity space environment.

Key Words: Diffusion, Molten Salts, Binary Systems, Melt and Solidification, Eutectics, Thermal Diffusion, Self-Diffusion, Interdiffusion, Soret Effect, Diffusion Coefficient, Thermal Gradient, Separation of Components, Buoyancy-Driven Convection, Volume Expansion, Porous Material, Wetting, Thermopotential Measurements, Density Difference, Sample Purity, Electrodes, Acceleration Effects, Accelerations/Vibrations Produced By Onboard Equipment

Number of Samples: six cells
Sample Materials: All cells contained the molten salt mixture: $AgI_{0.75}KI_{0.25}$. (silver iodide, potassium iodide)
(Ag*I*K*I*)
Container Materials: Thermodiffusion cells were made of impervious alumina. The cells were enclosed inside a stainless steel tube.
(Al*O*)

Experiment/Material Applications:
Improved theoretical knowledge of molten salts was sought to better enable dynamic calculations and simulations. Possible experimental applications include using thermodiffusion as a purifying process for various materials.

The Ag I-KI mixture was chosen because

"- first, one component is a silver salt, permitting to follow the electromotive force evolution versus time with convenient silver electrodes.

"- Secondly, the bonding forces are not purely ionic in the case of silver compounds so that one can assume the silver ions' heat of transport not to be neglected. This should produce a more important degree of separation.

"- Third, its melting point is not too high (250 ^{o}C - eutectic) which is an important factor in Space for a question of energetic consumption, specially for a long duration experiment (6 hours).

"- Fourth, for this eutectic composition the self-diffusion coefficient (Ag and K) are maximum, thus leading to a maximum interdiffusion coefficient in this mixture...." (5, p. 69)

References/Applicable Publications:

(1) Bert, J., Henry, D., Mellon, H., and Dupuy, J.: Space Thermal Diffusion Experiment in a Molten AgI-KI Mixture: Theoretical and Relation with in-situ Measurement Results. In Joint International Symposium on Molten Salts, Honolulu, Hawaii (USA), October 18-23, 1987.

(2) Bert, J., Moussa, I., Henry, D., and Dupuy, J.: Space Thermal Experiment in a Molten Salt AgI-KI Mixture. In BMFT/DFVLR Scientific Results of the D1 Symposium, Norderney, Germany, August 27-29, 1986, pp. 40-41. (post-flight; abstract only)

(3) Bert, J., Moussa, I., and Dupuy, J.: Space Thermal Diffusion Experiment in a Molten AgI-KI Mixture--Bernard Gourland Experiment. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 471-475.

(4) Bert, J. and Dupuy, J.: Measurement of the Soret Effect in Space. Naturwissenschaften, 73, Jahrgang Heft, July 1986, pp. 366-367. (post-flight)

(5) Bert, J. and Dupuy, J.: Space Thermal Diffusion Experiment in a Molten AgI/KI Mixture. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 69-71. (preflight)

(6) Bert, J., Henry, D., and Layani, P.: Space Experiment on Thermal Diffusion, Preparation and Theoretical Analysis. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 347-352. (experiment setup)

(7) Bert, J., Moussa, I., Henry, D., and Dupuy, J.: Space Thermal Diffusion Experiment in a Molten AgI-KI Mixture. pp. 48-56. (post-flight) <Note: Additional information on this publication is not available at this time.>

(8) Bert, J., Henry, D., Mellon, H., and Dupuy, J.: Space Thermal Diffusion Experiment in a Molten AgI-KI Mixture: Theoretical Convection Approach and Relation with in situ Measurement Results. Publication status unclear. <Note: The Principal Investigator reported that References (1) and (8) are the "same" paper (similar content and general significance) although the two do not contain exactly the same contents. It is believed that Reference (8) is the first version (draft) of Reference (1).>

(9) Input received from Principal Investigator J. Bert, July 1988 and July 1993.

(10) Hamacher, H., Merbold, U. and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium in Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements on D1)

(11) Bert, J., Moussa, I., Henry, D., and Dupuy, J.: Space Thermal Diffusion Experiment in a Molten AgI-KI Mixture. In Proceedings of the Norderney Symposium in Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 152-160. (post-flight)

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Experiment Origin: USA
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Payload Bay Materials Experiment Assembly (MEA-A2)
Processing Facility: Isothermal General Purpose Rocket Furnace (I-GPRF)
Builder of Processing Facility: Unknown, possibly The National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama

Experiment:
The Diffusion of Liquid Zinc and Lead (ME-GPRF 02)

Because of the extended solubility gap which exists in the Pb-Zn equilibrium system, severe limitations are placed on the alloy. Above the monotectic temperature, separation of the components due to gravity occurs rapidly (because of the large variation in density between the two materials). Therefore, alloys which have compositions between 0.9 wt.% and 99.5 wt.% Pb are not useful.

Processing in space (reduced gravity conditions) should allow the production of useful alloys within the desired composition range. It was expected that such an alloy could be produced under low-gravity conditions by (1) heating the desired Pb/Zn contained material above the consulate temperature, (2) holding the material at this temperature for a long enough period of time to allow homogenization by diffusion, and (3) solidifying the alloy.

The required homogenization time was not known since accurate diffusion constants had not been determined on Earth. Ground-based studies had indicated that low-gravity experiments were required to obtain this information. Thus, this Spacelab D1 experiment was designed to study the interdiffusion of zinc and lead under low-gravity conditions.

During the mission, two of the three containers available in the Spacelab Isothermal General Purpose Rocket Furnace (I-GPRF) were to be dedicated to the processing of two Pb-Zn samples (diffusion couples). <Note: A diagram in Reference (1) (released prior to flight) indicated that the samples were to be comprised of a rod of lead joined to a rod of zinc.> The furnace was mounted to the

MEA-A2 carrier, located in the shuttle cargo bay. One experiment was to be conducted just above the monotectic temperature of the system (440 ^{o}C) and the other just above the consulate temperature (820 ^{o}C). In each case, the temperatures were to be maintained for 40 minutes. After this period, the samples were to be rapidly cooled to lower than 315 ^{o}C. <The quench method was not discussed, but the furnace had the capability for gas quench.> The 40-minute soak time was established from (1) previous ground-based studies and (2) previously performed, low-gravity zinc self-diffusion experiments (see Ukanwa, Skylab SL-3 (this chapter)).

No post-flight publications could be located which discussed the results from this experiment. Reference (5) indicated that the experiment could not be operated. The exact reason for this difficulty was not discussed.

Key Words: Diffusion, Monotectic Compositions, Binary Systems, Metals and Alloys, Melt and Solidification, Interdiffusion, Liquid/Liquid Diffusion, Diffusive Mass Transfer, Liquid/Liquid Interface, Thermal Soak, Monotectic Temperature, Consulate Temperature, Separation of Components, Solubility Gap, Density Difference, Composition Distribution, Homogeneity, Solid/Liquid Interface, Quench Process, Sample Not Processed as Planned, Processing Difficulties

Number of Samples: two
Sample Materials: lead-zinc diffusion couples (Zn*Pb*)
Container Materials: unknown

Experiment/Material Applications:
An extended solubility gap exists in the Pb-Zn equilibrium system. This solubility gap severely restricts the use of this alloy system. Above the monotectic temperature, the two components separate because of their large density difference. Therefore, production of Pb-Zn alloys with compositions between 0.9 wt.% and 99.5 wt.% Pb have not been possible. Under low-gravity conditions, alloy compositions within this range should be possible.

References/Applicable Publications:

(1) Pond, R. B. and Winter, J. M.: The Diffusion of Liquid Zinc and Lead. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 76-77. (preflight)

(2) Materials Processing Experiments in Space: MEA-A2 Payload. Brochure available from Application Payload Projects, NASA/MSFC, Huntsville, Alabama. (MEA, preflight)

(3) General Purpose Rocket Furnace. In Microgravity Science and Applications Experiment Apparatus and Facilities, document developed by the Commercialization of Materials Processing in Space Group, Program Development Directorate, Marshall Space Flight Center, pp. 3-4. (processing facility) <Note: The year this document was published is unclear.>

(4) Pond, R. B., Sr. and Winter, J. W.: Pb-Zn Liquid-Metal Diffusion. In Microgravity Science and Applications Flight Programs, January-March 1987, Selected Papers, NASA TM-4069, Vol. 1, October 1988, pp. 511-521. (post-flight)

(5) Sahm, P. R.: Weightless Space Laboratory: The German Spacelab Mission D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 15-24. (post-flight)

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Experiment Origin: France
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt. Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, Materials Science Double Rack (MSDR)
Processing Facility: Gradient Heating Facility (GHF)
Builder of Processing Facility: French Space Agency (CNES), France

Experiment:
Thermodiffusion in Liquid Alloys (WL-GHF 07)

This Spacelab D1 experiment was the second in a series of investigations designed by Malmejac and/or Praizey to study thermomigration (Soret effect) in liquid alloys under low-gravity conditions (see Malmejac, Spacelab 1 (this chapter)). The specific objectives of the study were to (1) determine low-gravity thermomigration coefficients of tin alloys, and (2) improve the analysis of convective effects in the molten alloy system (see Reference (1) and Reference (6)).

During the Spacelab D1 mission, the Gradient Heating Facility (GHF) was used to process a total of twelve samples (see the Sample Materials section below). (The experimental setup and procedure were similar to the Spacelab 1 study (see Malmejac, Spacelab 1 ("Thermodiffusion in Tin Alloys")).) The samples, which had been placed within three zirconia shear cells, were subjected to a temperature range of 313 ^{o}C to 648 ^{o}C. After a soak period (chemical steady state), each sample was divided into six parts (while still molten) by rotating each of six portions within the shear cell a slightly different distance. The samples were then solidified, freezing the constituent content in each portion.

The following thermomigration coefficients were to be determined: (1) Sn_{112} in pure Sn, (2) Sn_{112} in an Sn-Ag alloy, (3) Ag_{109} in an Sn-Ag alloy, (4) Bi_{109} in an Bi-Sn alloy, and (5) Sn_{112} in Sn-Au. <Note: Although the Principal Investigator sent a list of the 12 samples processed, this list did not identify any Sn-Au samples. However, several of the publications listed below discussed the Sn-Au system.> The Sn-Ag alloys represented solutally destabilizing systems (on Earth, thermotransport carries the

heaviest elements upward) and the Sn-Bi and Sn-Au systems represented solutally stabilizing systems (on Earth, thermotransport carries the heaviest elements downward).

It was reported that "Operation of the rotation mechanism and behavior of the [shear] cells during the [low-gravity] experiment were not perfect because of the brittleness of the zircon.... This increased brittleness led to the fracture of disks and thus to the loss of certain samples. Thus, of the 12 samples used in the experiment, only 7 could be correctly processed. Fortunately the samples were distributed among the cells in such a manner that it was possible to study all the alloys...." (5, p. 173) However, it appears that the Sn-Bi alloy was analyzed with a "reduced statistical spread" compared to the other materials.

Post-flight, the component concentration of all of the samples was determined via neutron activation (except for the Bi sample which required chemical analysis). Reference (5), Reference (6), and Reference (7) included all the numerical values obtained as well the theoretical treatments presented. Please refer to these references for further information of these values/treatments.

When the results from this Spacelab D1 mission were compared with those from the Spacelab 1 experiment (see Malmejac, Spacelab 1, "Thermodiffusion in Tin Alloys" (this chapter)), the following conclusions were reported:

"-The experimental difficulties... and number of systems studied were such that, for each system, only a limited quantity of results was available,

"-activity measurement of ^{60}Co, originating from ^{59}Co, is easier than activity measurement of ^{113}Sn, originating from ^{112}Sn and ^{110}Ag originating from ^{109}Ag; bismuth can be analyzed only by chemical methods with an accuracy on the order of 10%,

"-but above all, the relative variation of concentrations in the temperature range studied is only a few percent for ^{112}Sn and ^{109}Ag and 30% for Bi, whereas it is 70% for Co." (7, p. 2392)

Finally, it was reported that for the Sn-Au system, "...if there is sufficient solute stabilization, measurements without convection disturbance can be obtained. However, for all destabilizing systems and for stabilizing systems in which a sufficient solute concentration cannot be achieved, the only way to obtain correct measurements is to perform microgravity experiments." (1, p. (12)312)

Key Words: Diffusion, Alloys, Binary Systems, Melt and Solidification, Thermal Diffusion, Thermomigration, Diffusion Coefficient, Soret Effect, Self-Diffusion, Diffusive Mass Transfer, Separation of Components, Density Difference, Thermal Gradient, Solutal Gradients, Thermal Soak, Convection, Buoyancy Effects Diminished, Concentration Distribution, Heat of Transport, Shear Cell, Solid/Liquid Interface, Hardware Malfunction, Processing Difficulties

Number of Samples: Twelve samples were processed in three cells.
Sample Materials: (1) Cell 1: one sample each of pure Sn, Sn-Ag 4%, Sn-Ag 0.4%, and Bi-Sn 1%; (2) Cell 2: one sample each of pure Sn, Sn-Ag 4%, Bi-Sn 1%, and Sn-Bi 2%; (3) Cell 3: one sample each of pure Sn, Sn-Ag 4%, Sn-Ag 0.4% 1%, and Sn-Bi 2%
(Sn*, Sn*Ag*, Bi*Sn*, Sn*Bi*)
Container Materials: Zirconia
(Zr*)

Experiment/Material Applications:
See Malmejac, Spacelab 1, "Thermodiffusion in Tin Alloys" (this chapter).

The Sn-Ag system was employed because ground-based studies had already been performed with this alloy. The Sn-Bi system had been studied during the "MEPHISTO" project and data on the system were also available.

References/Applicable Publications:
(1) Praizey, J. P.: Benefits of Microgravity for Measuring Thermomigration Coefficients in Liquid Metallic Alloys. In Advances in Space Research, Vol. 8, Number 12, 1988, pp. (12)305-(12)314. (post-flight; mainly theoretical treatment of convective effects)

(2) Praizey, J. P.: Thermomigration in Liquid Metallic. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 60. (post-flight)

(3) Praizey, J. P.: Thermomigration of Cobalt in Liquid Tin. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 77. (preflight)

(4) Praizey, J. P.: Thermomigration Dans Les Alliages Metalliques Liquides. In 6th European Symposium on Materials Science in Microgravity, Bordeaux, February 1987. (in French; post-flight)

(5) Praizey, J. P.: Thermomigration in Liquid Metallic Alloys; Measurements in Microgravity and Comparison with Results Obtained on the Ground. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 171-178. (post-flight)

(6) Malmejac, Y. and Praizey, J. P.: Thermomigration of Cobalt in Liquid Tin-Experiment 1 ES 320. In Proceedings of the 5th Symposium on Materials Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 147-152. (preflight; discusses Spacelab 1 study and theoretical analysis of convective effects)

(7) Praizey, J. P.: Benefits of Microgravity for Measuring Thermotransport Coefficients in Liquid Metallic Alloys. In International Journal of Heat and Mass Transfer, Vol. 12, Number 12, 1989, pp. 2385-2401. (post-flight; Spacelab 1 and Spacelab D1 results as well as theoretical treatments)

(8) Input received from Principal Investigator J. P. Praizey, July 1993.

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Co-Investigator(s): Unknown
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Experiment Origin: Federal Republic of Germany
Mission: STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
Launch Date/Expt Date: October 1985
Launched From: NASA Kennedy Space Center, Florida
Payload Type: STS Spacelab Facility, M E D E A Double Rack
Processing Facility: Gradient Furnace with Quench (GFQ)
Builder of Processing Facility: Dornier Friedrichshafen, Germany

Experiment:
Aluminum/Copper Phase Boundary Diffusion - Solid-Liquid Interface Diffusion (MD-GFQ 01)

This Spacelab D1 experiment was designed to study the influence of convection on the solidification of metallic alloys. The specific objectives of the investigation were to determine thermal convective influences on (1) the mass diffusion in the melt ahead of a solid-liquid interface (in an AlCu alloy), (2) the stability of this interface, and (3) the morphology of an interface in an AlSi alloy.

During the Spacelab D1 mission, five contained samples (three Al-7.0 wt.% Si alloys and two Al-0.3 wt.% Cu alloys) were processed in the Gradient Furnace with Quench (GFQ). The samples were processed such that the influence of double-diffusive convection and Marangoni convection was negligible. Four thermocouples were placed through the center of all samples for thermal data acquisition. After directional solidification procedures, the specimens were quenched to stop the solidification front. The quench was initiated by the application of a water spray (followed by a mixture of He and Ar to the crucible surface (see Reference (3) for details)). Samples were also processed on Earth for comparison purposes.

Post-flight examination of the samples consisted of metallographic analysis and microsonde measurements for concentration profile determination. It was reported that for the AlCu alloys, the diffusion boundary layer of the low-gravity material was twice as thick when compared to the boundary layer of 1-g processed samples (same crystallization condition). (A 9 mm boundary layer was indicated for one of the space-processed specimens (see Reference (3) for quantitative and theoretical treatments of these results).)

The quenched solidification front of one (low-g) AlCu sample was examined. Reportedly, the interface between the solid and quenched liquid zone was unstructured and smooth. This result indicated that the criteria described by G/v (where G = sample temperature gradient and v = solidification velocity) for a smooth solidification front appears to have a thermal convection term which has not been considered (see Reference (3) for details).

The influence of convection on dendrite arm coarsening was examined in the AlSi samples. It was determined that coarsening was lower in the 1-g processed samples when solidified with a thermal gradient of approximately 16 K/mm and a solidification velocity of about 5 mm/min. However, if the velocity was increased to about 8 mm/min (thermal gradient = 16 K/mm), the dendrite arm coarsening in the 1-g and low-gravity processed samples was nearly identical. Therefore, it appeared that convection only influenced the dendrite arm spacing below a certain solidification front velocity. (Theoretical treatment of the convection influences on dendrite arm spacing can be located in Reference (6).)

No further information concerning the analysis of the flight samples could be located at this time.

Key Words: Diffusion, Metals, Binary Systems, Metals and Alloys, Melt and Solidification, Directional Solidification, Diffusion Boundary, Diffusive Mass Transfer, Double Diffusive Convection, Marangoni Convection, Thermal Convection, Thermosolutal Convection, Thermal Gradient, Solidification Rate, Growth Rate, Boundary Layer, Solid/Liquid Interface, Interface Stability, Solidification Front Physics, Concentration Distribution, Dendrites, Dendritic Arm Spacing, Dendritic Structure, Quench Process

Number of Samples: five
Sample Materials: Two samples: Al-0.3 wt.% Cu; three samples: Al-7.0 wt.% Si
(Al*Cu*, Al*Si*)
Container Materials: unknown

Experiment/Material Applications:
The specific reasons why AlCu and AlSi alloys were selected for the experiments were not detailed in the available publications.

References/Applicable Publications:
(1) Tensi, H. M. and Schmidt, J.: Influence of Thermal Gravitational Convection on Solidification Processes. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986. p. 39. (post-flight)

(2) Tensi, H. M.: Solid Liquid Interface Diffusion. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 97-98. (preflight)

(3) Tensi, H. M. and Schmidt, J. J.: Influence of Thermal Gravitational Convection on Solidification Processes. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 216-222. (post-flight)

(4) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements on D1)

(5) Tensi, H. M. and Mackrodt, C.: Possibilities of Investigating the Crystallization Parameters During Unidirectional Solidification. Appl. Microgravity Tech. II (1989), 2, pp. 68-74. (post-flight)

(6) Tensi, H. M.: Influence of Microgravity on the Morphology of the Directionally Solidified Front in an AlSi Alloy. Metallurgical Transactions A, Vol. 19A, November, 1988, pp. 2681-2686. (post-flight; discusses results from AlSi samples only)

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Co-Investigator(s): Urbanek, J. (2), Hehenkamp, Th. (3)
Affiliation(s): (1) During TEXUS 16: Universität Göttingen, Federal Republic of Germany; Currently: Hoechst AG, Frankfurt, Germany; (2,3) Institut für Metallphysik, der Universität Göttingen, Germany

Experiment Origin: Federal Republic of Germany
Mission: TEXUS 16
Launch Date/Expt. Date: November 1987
Launched From: ESRANGE, Kiruna, Northern Sweden
Payload Type: Sounding Rocket Experiment
Processing Facility: TEXUS Experiment Module TEM 01-2
Builder of Processing Facility: MBB-ERNO, Bremen, Germany

Experiment:
Diffusion of Ni in CuAl and CuAu Alloys

During this sounding rocket experiment, the diffusion of nickel in copper alloys was to be investigated.

Reportedly, shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including the TEM 01-2 module.

No further information concerning the experimental objectives or equipment setup of this investigation could be located at this time.

Key Words: Diffusion, Metals and Alloys, Diffusive Mass Transfer, Interdiffusion, Acceleration Effects, Payload Survivability, Rocket Failure, Payload Recovery System Failure

Number of Samples: unknown
Sample Materials: nickel, copper-aluminum, copper-gold (Ni*Cu*Al*, Ni*Cu*Au*)
Container Materials: unknown

Experiment/Material Applications: Unspecified, although typical applications are cited throughout this chapter.

References/Applicable Publications:

(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 109-111. (in German; post-flight)

(2) Input received from Experiment Investigator, June 1991.

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Principal Investigator(s): Fiske, M. R. (1)
Co-Investigator(s): None
Affiliation(s): (1) Teledyne Brown Engineering, Huntsville, Alabama

Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Solid-Liquid Diffusion

Organic systems are often used to model metal alloy systems because (1) such systems normally melt at significantly lower temperatures than metal alloy systems, (2) at these low temperatures, visual recording of the solidification can take place, (3) the phase diagrams of such systems can be remarkably similar to metal alloy systems, and (4) by employing these diagrams, interpretation/extrapolation/prediction of metal alloy system microstructural features is possible.

One of the primary goals of solidification research is understanding the phenomena occurring at the growth front. "These phenomena are governed by convection and diffusion in the liquid ahead of the front. By removing the convection term (in a low gravity field), a more detailed understanding of diffusion-controlled growth will result." (7, p. 1)

The objective of this Consort 1 experiment was to measure the low-gravity diffusion of a liquid (ethanol) into a solid (succinonitrile). It was expected that measurement of the diffusion coefficient would "...lead to better definition of this system as well as metal alloy systems without having to build expensive, high-temperature furnaces." (7, p. 1)

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1)); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams

(including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments), the apparatus was also used to (1) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (2) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (1) aligned or (2) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1 (Chapter 16)). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1 (all in Chapter 1)). This experiment was allotted two "Type 2" test wells. Discussion of this specific well-type is detailed here.

Each "Type 2" test well provided the investigator with one experimental opportunity (data point). The well-type used two sample wells, one on the top block and one on the bottom block. Prior to flight, the top well of each well-type was filled with ethanol and the bottom well of each well-type was filled with succinonitrile. The blocks were then joined together such that the wells in the upper block were purposely misaligned with the wells in the lower block.

Once the rocket had been launched and the low-gravity phase had been achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. The MDA experiments were performed in an ambient temperature environment and thus, the succinonitrile was in the solid phase. Once the wells were in contact, material diffusion was realized across the solid-liquid interface. Just prior to the termination of the low gravity rocket phase, the upper block again moved

right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. However, fluid leakage from wells associated with another experiment within the MDA resulted in contamination of the test wells allotted to this solid/liquid diffusion investigation. The principal investigator reported that this contamination invalidated the post-flight analyses.

No further information concerning this experiment is expected to be made available.

Key Words: Diffusion, Solid/Liquid Diffusion, Diffusion-Controlled Growth, Solid/Liquid Interface, Model Materials, Organic Systems, Solidification Front Physics, Interface Physics, Buoyancy-Driven Convection, Liquid Leakage, Contamination Source

Number of Samples: two
Sample Materials: Top wells: ethanol (135-μl); bottom wells: succinonitrile (135-μl)
Container Materials: inert material

Experiment/Material Applications:
These experiments are applicable to metal alloy systems which exhibit similar phase behavior. If these systems can be better understood, new microstructural features and/or improved physical and mechanical properties of the alloys may result.

References/Applicable Publications:
(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Information supplied by ITA detailing Top and Bottom Well Contents, dated February 1989. (provided by ITA to C. A. Winter, 1/91.)

(5) Correspondence from M. R. Fiske to J. Cassanto (ITA), 10/18/88. (provided by ITA to C. Winter 1/91; preflight, experiment applications)

(6) Input received from Principal Investigator M. R. Fiske, May 1991 and July 1993.

(7) Correspondence from M. R. Fiske to J. Cassanto (ITA) 4/13/89. (provided by Fiske to C. Winter 5/91; post-flight, no results, details experiment applications/objectives)

(8) Correspondence from M. R. Fiske to J. Cassanto (ITA) 4/14/89. (provided by Fiske to C. Winter 5/91; post-flight, no results, details experiment applications/objectives)

(9) Frohberg, G., et al.: Self-Diffusion of Sn^{112} and Sn^{124} in Liquid Tin. In Proceedings of the 5th European Symposium on Materials Sciences Under Microgravity, Schloss-Elmau, November 5-7, 1984, p. 210. (related research; reference used to help design experiment)

(10) "Transparent Analogs for Alloy Phase Studies," NASA Tech Brief, Number MFS-27109. (related research)

(11) Schaefer, R. J. and Coriell, S. R.: Convective and Interfacial Instabilities During Solidification of Succinonitrile Containing Ethanol. In Materials Processing in the Reduced Gravity Environment of Space, Guy Rindone, editor, 1982, p. 479. (related research)

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Co-Investigator(s): Unknown
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Experiment Origin: USA
Mission: Consort 1 (Starfire Rocket)
Launch Date/Expt. Date: March 1989
Launched From: White Sands Missile Range, New Mexico
Payload Type: Sounding Rocket Experiment
Processing Facility: Materials Dispersion Apparatus (MDA) Minilab
Builder of Processing Facility: Instrumentation Technology Associates, Inc., Exton, Pennsylvania

Experiment:
Turbulent Mixing and Dye Diffusion

The specific objective of this Consort 1 turbulent mixing and dye diffusion experiment was to determine the "mass transfer baseline for diffusion." (3, viewgraphs)

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1)); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (three other experiments, Chapters 1 and 2); Vera, Consort 1 (two experiments, Chapter 1))). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (11 experiments) the apparatus was also used to (1) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (2) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (1) aligned or (2) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the

other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1 (Chapter 16)). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1 (all in Chapter 1)). This experiment was allotted five "Type 2" test wells. Discussion of this specific well-type is detailed here.

Each "Type 2" test well provided the investigator with one experimental opportunity. The well-type used two sample wells, one on the top block and one on the bottom block. Prior to the rocket flight, the well in the upper block of each well-type was filled with water and the well in the lower block of each well-type was filled with trypan blue dye, 2% or 4% in water. <Note: Reference (6) indicated that 2% dye in water was used while Reference (2) indicated that 4% dye in water was used.> The blocks were then joined together such that the well in the upper block was purposely misaligned with the well on the lower block. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity rocket phase, the upper block again moved right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Optical density measurements of each well-pair performed after the rocket flight illustrated the amount of dye which had diffused during the low gravity phase of the rocket flight.

Very little discussion of results could be located at this time. However, it was reported that "...diffusion of trypan blue from a water solution into... pure water, occurs at the same rate in low

gravity as on earth. This observation served as the baseline for approximately 70 other samples on the MDA and affirmed that solute transport was due primarily to diffusion plus the mixing that occurs at time=0 measured on the ground." (5, p. 7)

A short report detailed in Reference (5) appears to couple results of this experiment with two others by Todd (see also Todd, Consort 1, Capillary Flow (Chapter 2); Todd, Consort 1, Phase Rearrangement (Chapter 1)): "Steep surface-tension gradients were established in two types of transport experiments involving two fully-enclosed liquids with an interface between them. When the two liquids were immiscible aqueous solutions [Phase Rearrangement Experiment], no evidence of capillary flow or reorientation of the phases was obtained during the low gravity period. When the two liquids were miscible aqueous solutions (one with detergent [Capillary Flow Experiment] and one without [Turbulent Mixing Experiment]) there was also no capillary flow. This last observation could not be made definitely on the ground, where transport was dominated by convection, owing to the similar densities of the two solutions." (5, p. 7)

No further information discussing specific experiment objectives or results could be located at this time.

Key Words: Diffusion, Diffusive Mass Transfer, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Aqueous Solutions, Liquid Mixing, Turbulent Flow, Buoyancy-Driven Convection, Surface Tension, Contamination Source, Liquid Leakage

Number of Samples: five
Sample Materials: Top wells: water; bottom wells: trypan blue, 2% or 4% in water. (Reference (2) indicates that there was 4% dye in water; Reference (6) indicates that there was 2% dye in water.)
Container Materials: inert material

Experiment/Material Applications:
A brief note in Reference (3) indicated that this investigation had applications in the area of baseline diffusion experiments.

No further information discussing the applications of this investigation could be found.

References/Applicable Publications:

(1) Hertz, R. M. and Todd, P.: Turbulent Mixing and Diffusion of Solutes. Information sent by ITA, April 3, 1990 to C. Winter (NASA). (post-flight)

(2) Letter from P. Todd (National Institute of Standards and Technology) to J. C. Cassanto (Instrumentation Technology Associates), dated May 9, 1989, discussing analysis efforts completed to date. (Information provided by Instrumentation Technology Associates (ITA) to C. A. Winter, 1/91) (post-flight)

(3) Cassanto, J. M.: Overview/Summary of Results From the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(4) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(5) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(6) Information supplied by ITA detailing top and bottom well contents, dated February 1989. (provided by ITA to C. A. Winter 1/91)

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13. ABSTRACT *(Maximum 200 words)*

An electronic data base identifying over 800 fluids and materials processing experiments performed in a low-gravity environment has been created at NASA Marshall Space Flight Center. The compilation, called MICREX (MICrogravity Research Experiments), was designed to document all such experimental efforts performed (1) on U.S. manned space vehicles, (2) on payloads deployed from U.S. manned space vehicles, and (3) on all domestic and international sounding rockets (excluding those of China and the former U.S.S.R.). Data available on most experiments include (1) principal and co-investigators, (2) low-gravity mission, (3) processing facility, (4) experimental objectives and results, (5) identifying key words, (6) sample materials, (7) applications of the processed materials/research area, (8) experiment descriptive publications, and (9) contacts for more information concerning the experiment. This technical memorandum (1) summarizes the historical interest in reduced-gravity fluid dynamics, (2) describes the experimental facilities employed to examine reduced gravity fluid flow, (3) discusses the importance of a low-gravity fluids and materials processing data base, (4) describes the MICREX data base format and computational World Wide Web access procedures, and (5) documents (in hard-copy form) the descriptions of the first 600 fluids and materials processing experiments entered into MICREX.

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